AD NUMBER

AD801858

LIMITATION CHANGES

TO:

Approved for public release; distribution is unlimited.

FROM:

Distribution authorized to U.S. Gov't. agencies and their contractors; Critical Technology; AUG 1966. Other requests shall be referred to Air Force Materials Laboratory, ATTN: MAAM, Wright-Patterson AFB, OH 45433.

AUTHORITY

AFML ltr dtd 12 Jan 1972

AFML-TR-66-223

PHYSICAL PROPERTIES OF REFRACTORY MATERIALS

S. A. Bortz
IIT Research Institute

TECHNICAL REPORT NO. AFML-TR-66-223
August 1966

Air Force Materials Laboratory
Research and Technology Division
Air Force Systems Command
Wright-Patterson Air Force Base, Ohio

NOTICES

When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related Government procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data, is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

Copies of this report should not be returned to the Research and Technology Division unless return is required by security considerations, contractual obligations, or notice on a specific document.

AFML-TR-66-223

PHYSICAL PROPERTIES OF REFRACTORY MATERIALS

S. A. Bortz
IIT Research Institute

TECHNICAL REPORT NO. AFML-TR-66-223
August 1966

Air Force Materials Laboratory
Research and Technology Division
Air Force Systems Command
Wright-Patterson Air Force Base, Ohio

FOREWORD

This report was prepared by the Ceramics Research Division of IIT Research Institute, Chicago, Illinois, under Contract No. AF 33(615)-3028. The contract was initiated under Project No. 7381, "Materials Application," Task No. 738106, "Design Information Development." The work was administered under the direction of Marvin Knight, of the Materials Information Branch (MAAM), Research and Technology Division, Air Force Materials Laboratory, Wright-Patterson Air Force Base, Ohio.

This report covers work conducted from 1 May 1965 to 1 May 1966.

S. A. Bortz, Senior Engineer, Ceramics Research, was project engineer. Others who cooperated in the research were C. Connors, Assistant Engineer; J. Hedge, Research Engineer; V. Hill, P. Herman and P. Janson.

Detailed data for the program are recorded in IITRI Logbooks Nos. C 16489 and C 16815. This report has been given the internal designation IITRI-G-6001-12.

Manuscript of this report was released by the author in May 1966 for publication as an RTD Technical Report.

This technical report has been reviewed and is approved.

D. A. SHINN
Chief, Materials Information Branch
Materials Applications Division
Air Force Materials Laboratory

ABSTRACT

The purpose of this program is to characterize and correlate the physical and mechanical properties of JTA graphite and, from these data, determine its critical engineering properties.

Three JTA billets were studied, two of 7 in. diameter by 6 1/2 in. long, and one 14 in. diameter by 7 in. long. These were examined by Carbon Products Division of Union Carbide Corporation for gross flaws and differences in density, by radiographic methods. The material was found to be acceptable within the limitations, as provided in the procurement specifications. The densities of the three billets are above 3.00 g/cc. Metallographic, x-ray, and microprobe analyses confirm the presence of graphite, ZrB_2, and βSiC. Metallographic examination indicates only a slight variation in distribution of the three principal elements of JTA graphite. The ZrB_2 and βSiC content of the edges of the billet appear to be somewhat less than that in the center.

Mechanical measurements indicate that JTA graphite is anisotropic; the "with" grain orientation is about twice as strong as the "across" grain orientation. Size effect studies indicate that a minimum 1/4 in. cross section dimension is adequate for determining average mechanical properties. Studies were made using both flexure and tension measurements. The mechanical strength of the three billets appears to be the same.

The specific heat of the three billets was found to be the same.

Temperature and oxygen pressure have a great effect on the oxidation resistance of JTA graphite. The material appears to be more resistant at the higher temperatures.

CONTENTS

ILLUSTRATIONS

ILLUSTRATIONS (Continued)

TABLES

I. INTRODUCTION

This report consists of a summary of work accomplished during the period of May 1965 to May 1966, under Contract No. AF 33(615)-3028, which is directed toward obtaining property data for JTA graphite. Delays in obtaining material, due to manufacturing problems, have caused the program to proceed at a very low rate during this contract period. The work reported on is approximately equivalent to that originally planned for the first quarter of the program.

Nose cone, rocket nozzle, and leading edge applications under extreme environmental conditions require materials which will withstand severe thermal and mechanical conditions. Attention has been focused on the refractory composite materials because of their potential high strength at elevated temperatures, thermal insulation, and oxidation resistance. However, most of these materials often exhibit a wide variability in properties used in the design of structures required to resist extreme environments. This investigative program is designed to correlate the physical and mechanical parameters of composite refractory oxidation-resistant material, JTA graphite.

Considerable emphasis has been placed on determining the uniformity of JTA from billet to billet and from one size billet to another. Data have been obtained from two 7 in. diameter by 6 1/2 in. long JTA billets and one 14 in. diameter by 7 in. long billet. The information developed during this program is being used to analyze and compare data obtained from previous programs.

II. SUMMARY

Not enough data have been obtained to draw any definite conclusions; however, the following statements appear to be valid from the limited data available:

1. There are no gross flaws or density differences through the billets. The material under study consists of graphite, ZrB_2, and βSiC.

2. The three billets appear to have roughly the same composition with only a slight microstructural variability throughout the bulk material.

3. The densities are above the specified 3.00 g/cc and appear to be uniform throughout the bulk material.

4. A 1/4 in. major cross-sectional dimension is adequate for determining the average JTA graphite mechanical properties.

5. The JTA graphite is highly anisotropic, and the failure modes for the "with" grain and "across" grain orientations appear to be very dissimilar. The former has a statistical failure range similar to oxide ceramics, while the latter is "deterministic" and can be treated as a metal.

6. The temperature and oxygen pressure have a profound effect on the oxidation resistance of JTA. The material appears to be more resistant at the higher temperatures.

7. The enthalpies of material from the three billets measured are the same.

Table I provides a summary of the mechanical property data presently available.[1] These data can be compared to those shown in Table II. The room-temperature data obtained to date appears comparable to some of the data previously obtained.

Experimental work is being continued to obtain the remainder of the data as outlined in Section III, Material Characterization.

III. MATERIAL CHARACTERIZATION

JTA graphite is manufactured by Carbon Products Division of Union Carbide Corporation. This grade is a multiple-phase composite material[2] containing 48 wt% carbon 35 wt% zirconium, 8 wt% boron, and 9 wt% silicon, which is hot-pressed in an induction-heated graphite mold. Carbon Products Division designed this material for good oxidation resistance and mechanical strength at high temperatures. Material characterization makes use of metallographic techniques, electron microprobe analyses, and x-ray analyses, and relates these findings with the following properties:

1. Density
2. Oxidation resistance
3. Porosity
4. Tensile strength
5. Compressive strength
6. Flexural strength
7. Elastic properties (Young's modulus and Poisson's ratio)
8. Creep
9. Thermal shock resistance
10. Thermal expansion
11. Emittance
12. Specific heat
13. Thermal diffusivity

Table I

PREVIOUS DATA ON JTA GRAPHITE[1]
(Room temperature, 14 in. diameter billets)

Property	With Grain				Across Grain			
	No. of Tests	Avg.	Error (ν),%	Billet No.	No. of Tests	Avg.	Error (ν),%	Billet No.
Bulk density, g/cc	22	2.966	23	1	8	3.028	1.1	1
	21	2.854	3.5	2				
Coefficient of expansion, in/in/°C:								
RT to 1500°C	2	5.6		1,2	2	6.0		1
1500°-2000°C	2	5.6		1,2	2	11.0		1
Tensile strength, psi	4	10,010	7.0	1				
Tensile elastic modulus, 10^6 psi (from Ref. 4)		5.6				3.6		
Flexure strength, psi	5	17,750	4.7	1	5	9,592	5.2	1
	5	12,784	5.6	2				
Flexure elastic modulus, 10^6 psi	5	9.1		1	5	4.95		1
	5	7.1		2				
Compressive strength, psi	3	15,330	2.6	1	5	22,160	8.7	1
	5	12,592	11.2	2				
Compressive elastic modulus, 10^6 psi	5	2.9		1	5	3.6		1

Table II

PROPERTIES OF JTA GRAPHITE (ROOM TEMPERATURE)

Property	With Grain			Across Grain		
	No. of Tests	Avg.	Error (ν), %	No. of Tests	Avg.	Error (ν), %
Bulk density, g/cc						
Billet 7-F-12	20	3.091	0.68			
Billet 7-F-14	20	3.077	0.74			
Billet 14-G-1	20	3.065	1.60			
Tensile strength, psi	5	11,970	7.35	5	6000	3.80
Flexure strength, psi	60	17,850	14.87	60	8940	9.73
Flexure elastic modulus, 10^6 psi	60	11.0	21.14	60	4.5	26.06

The interrelationships of these measurements are analyzed--for example, mechanical and thermal properties with thermal-shock resistance, or oxidation resistance with mechanical and thermal properties.

The billets were sectioned and radiographed for cracks and voids in excess of 0.03 in. in major dimensions by Carbon Products before they were sent to IITRI for use on this program. Each billet was identified as follows: 7-F-12, 7-F-14, and 14-G-1. The first number identifies the diameter of the billet, the letter is the code for the pressing series, and the last number identifies the billet for a particular pressing series.

Figure 1 shows how the billets were cut for radiographic examination. Each piece was given three exposures: 4 min at 265 kv, 3 1/2 min at 240 kv, and 2 1/2 min at 200 kv. A medium speed film, type AA, was used for this work. The exposures are shown in Figs. 2, 3, 4, 5, and 6. Examination of these negatives reveals that the billets appear to be a heterogeneous mixture. No major flaw areas can be distinguished, nor can large differences in density be detected.

Samples were cut from each billet as shown in Fig. 7 for the 7 in. billets and Fig. 8 for the 14 in. billet. The material cut from these areas was used for metallographic examination, microprobe, and x-ray analyses.

A. Powder X-Ray Analysis

Powder x-ray patterns have been completed for billet 7-F-12. The results of this analysis are listed in Table III. The major constituents were found to be graphite, zirconium diboride (ZrB_2), and traces of β silicon carbide (βSiC). Carbon Products[2] reports that graphite, zirconium diboride, and α silicon carbide (αSiC) should be present in JTA graphite. The difference in the SiC structure is probably due to changes in fabrication procedures between the early experimental material and the material used for this program. The βSiC is the low-temperature cubic phase of this compound which begins to convert to the hexagonal phase at 2150°C. Assuming that the raw material consisted of powders of carbon, zirconium diboride, and silicon, βSiC will begin to form at temperatures above 2150°C and will be completely transformed to αSiC between 2150° and 2300°C.[3] If the material is very slowly cooled from 2300°C to room temperature, the α will revert back to βSiC. However, this process usually will not occur under normal cooling procedures. The inference is that the JTA graphite under study in this program was hot-pressed at a temperature below 2200°C.

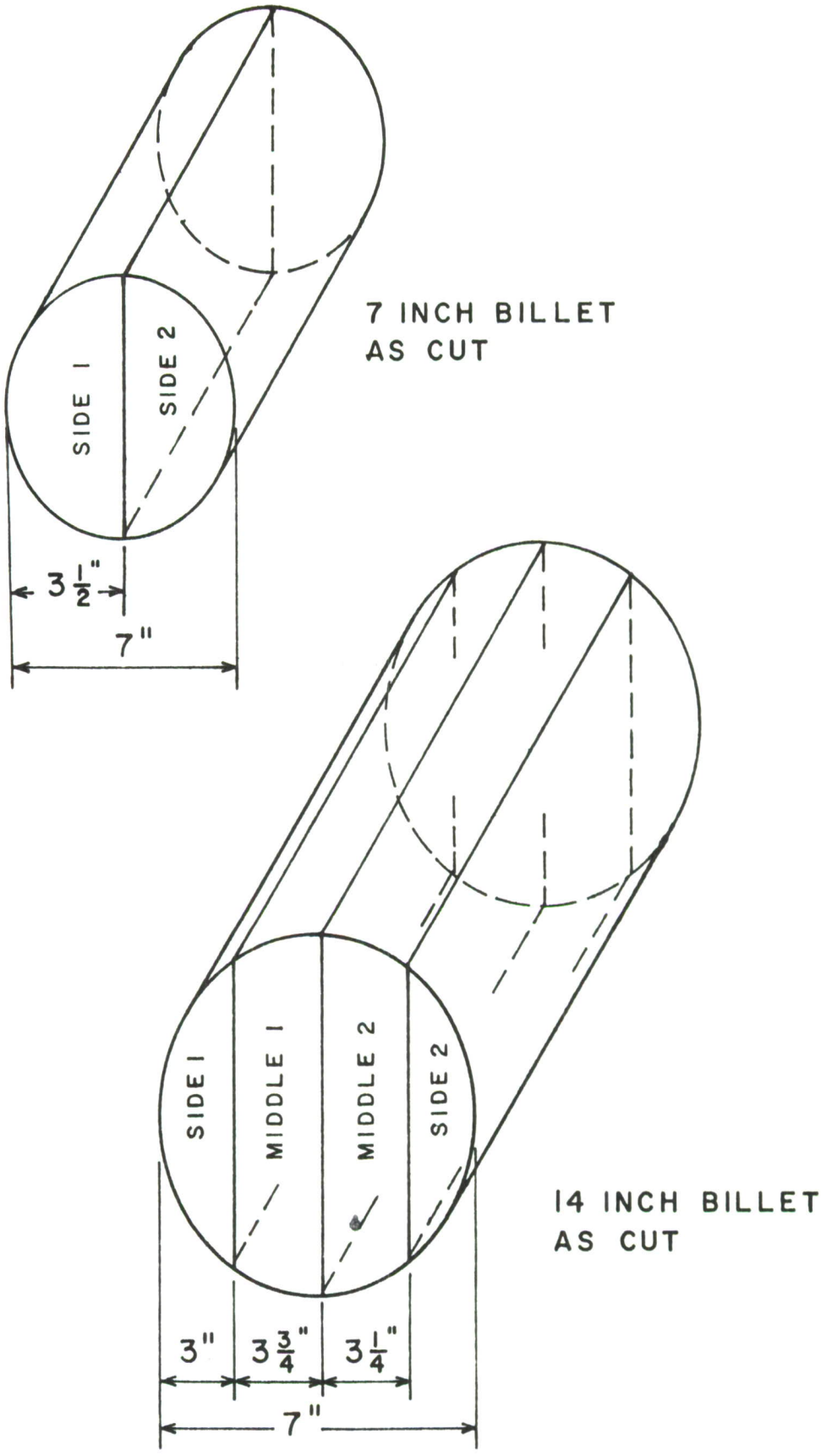
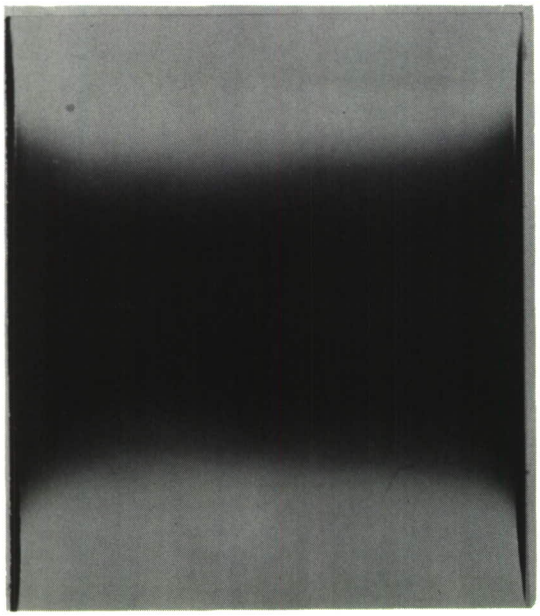
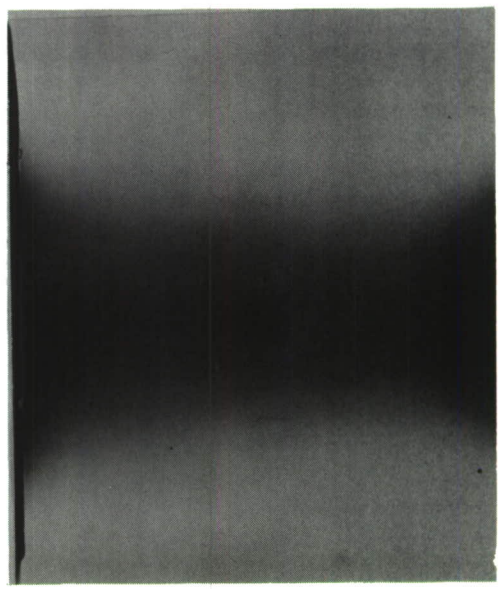


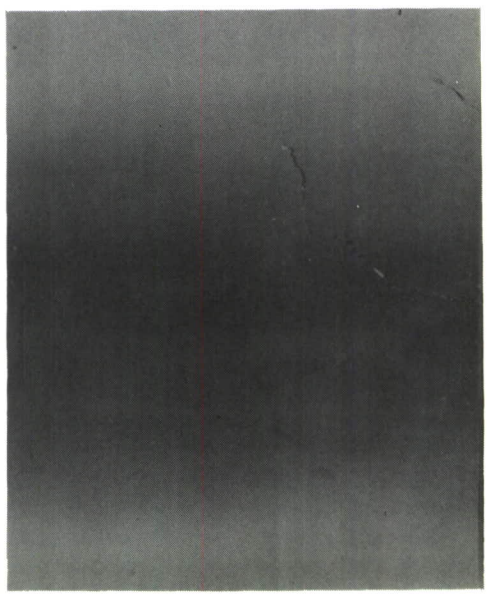
Fig. 1 - SECTIONING OF JTA BILLETS
FOR RADIOGRAPHIC ANALYSIS



4 MIN. 265 KV
EXPOSURE

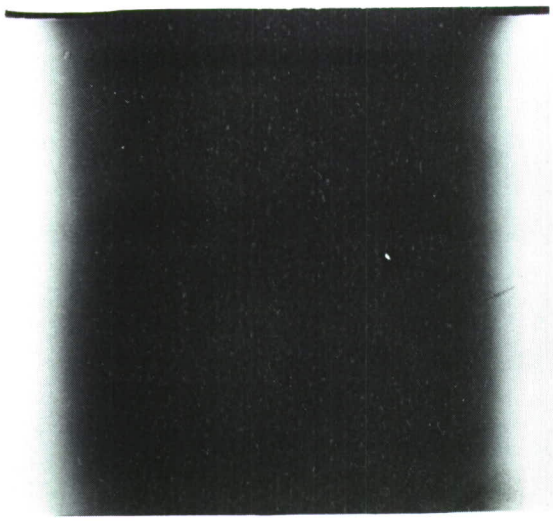


3 1/2 MIN. 240 KV
EXPOSURE

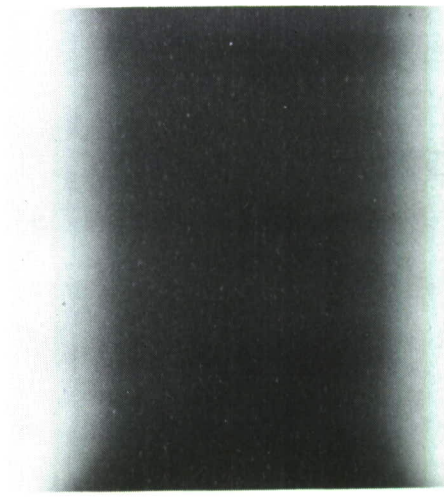


3 1/2 MIN, 240 KV
EXPOSURE

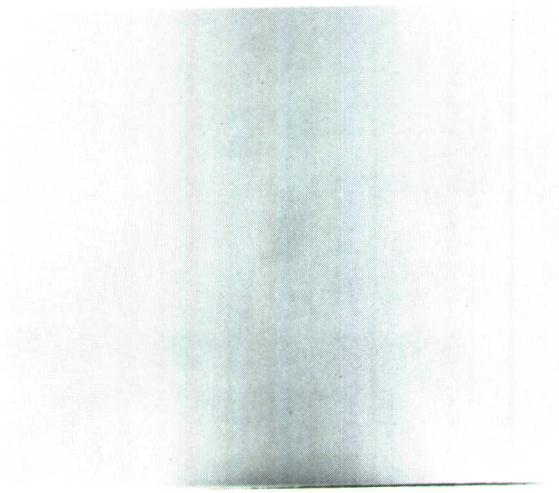
Fig. 2 - RADIOGRAPHS OF BILLET 7-F-12 (SIDE 1)



4 MIN, 265 KV
EXPOSURE

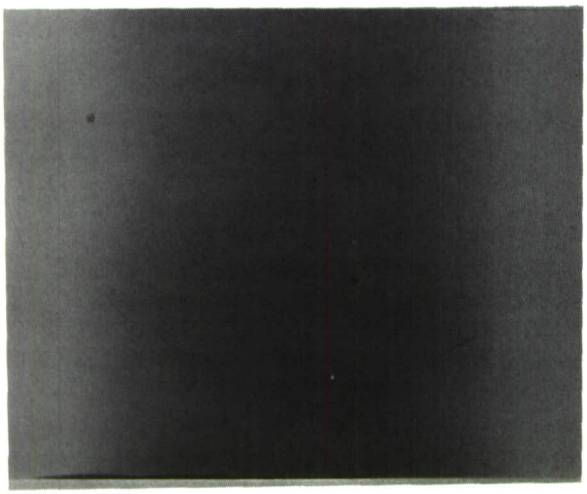


3 1/2 MIN, 240 KV
EXPOSURE

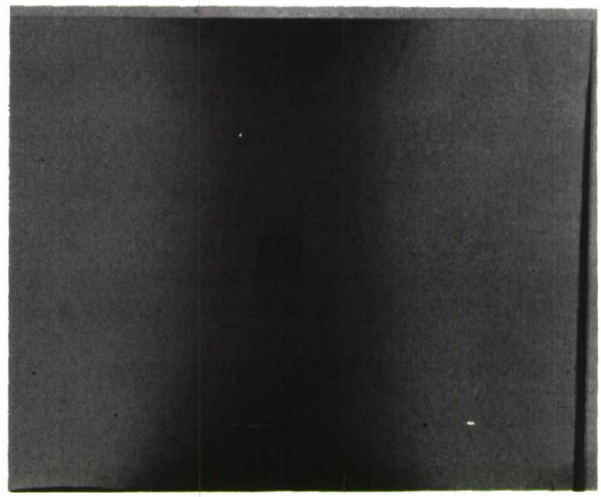


2 1/2 MIN, 200 KV
EXPOSURE

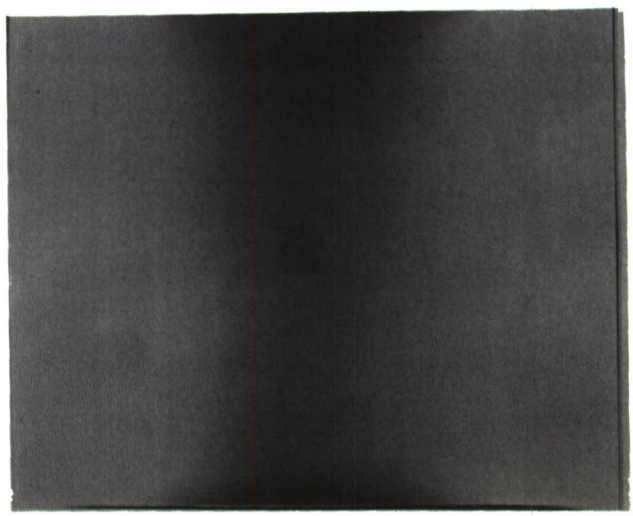
Fig. 3 - RADIOGRAPHS OF BILLET 7-F-12 (SIDE 2)



4 MIN, 265 KV
EXPOSURE

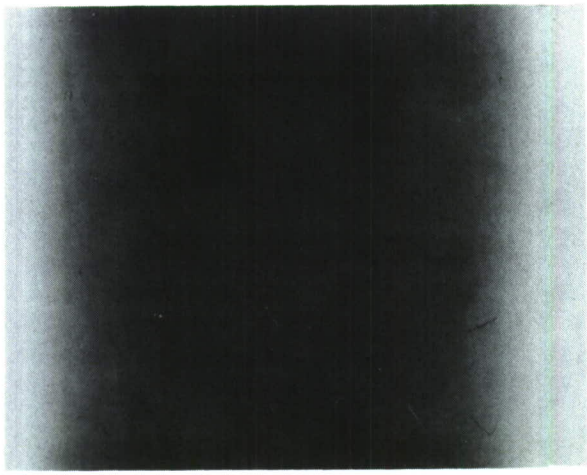


3 1/2 MIN, 240 KV
EXPOSURE

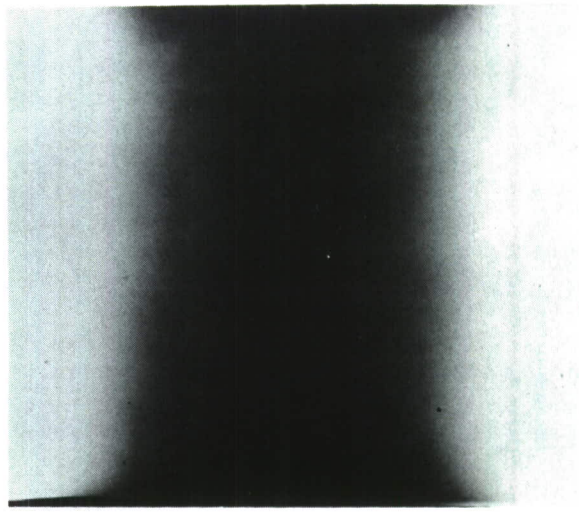


2 1/2 MIN, 200 KV
EXPOSURE

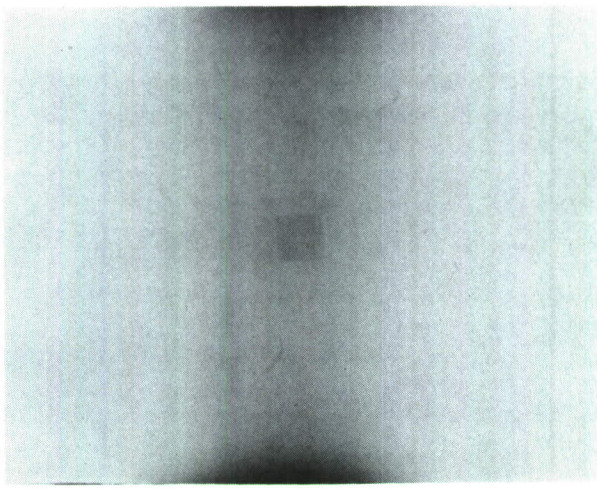
Fig. 4 - RADIOGRAPHS OF BILLET 7-F-14 (SIDE 1)



2 1/2 MIN, 200 KV
EXPOSURE

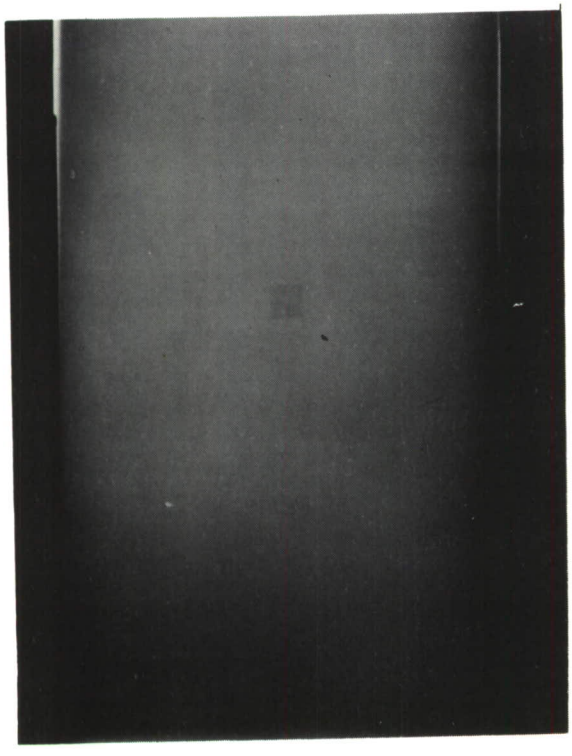


3 1/2 MIN, 240 KV
EXPOSURE

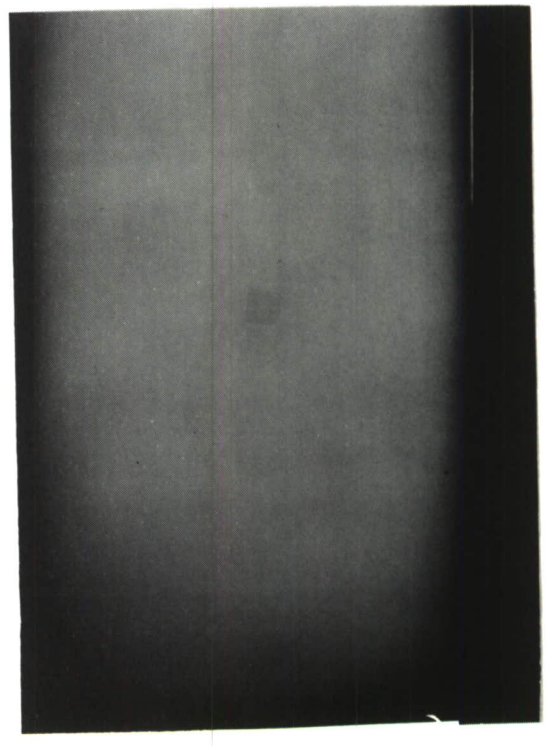


4 MIN, 265 KV
EXPOSURE

Fig. 5 - RADIOGRAPHS OF BILLET 7-F-14 (SIDE 2)



4 MIN, 265 KV
EXPOSURE



3 1/2 MIN, 240 KV
EXPOSURE

2 1/2 MIN, 200 KV
EXPOSURE

Fig. 6 - RADIOGRAPHS OF BILLET 14-G-1

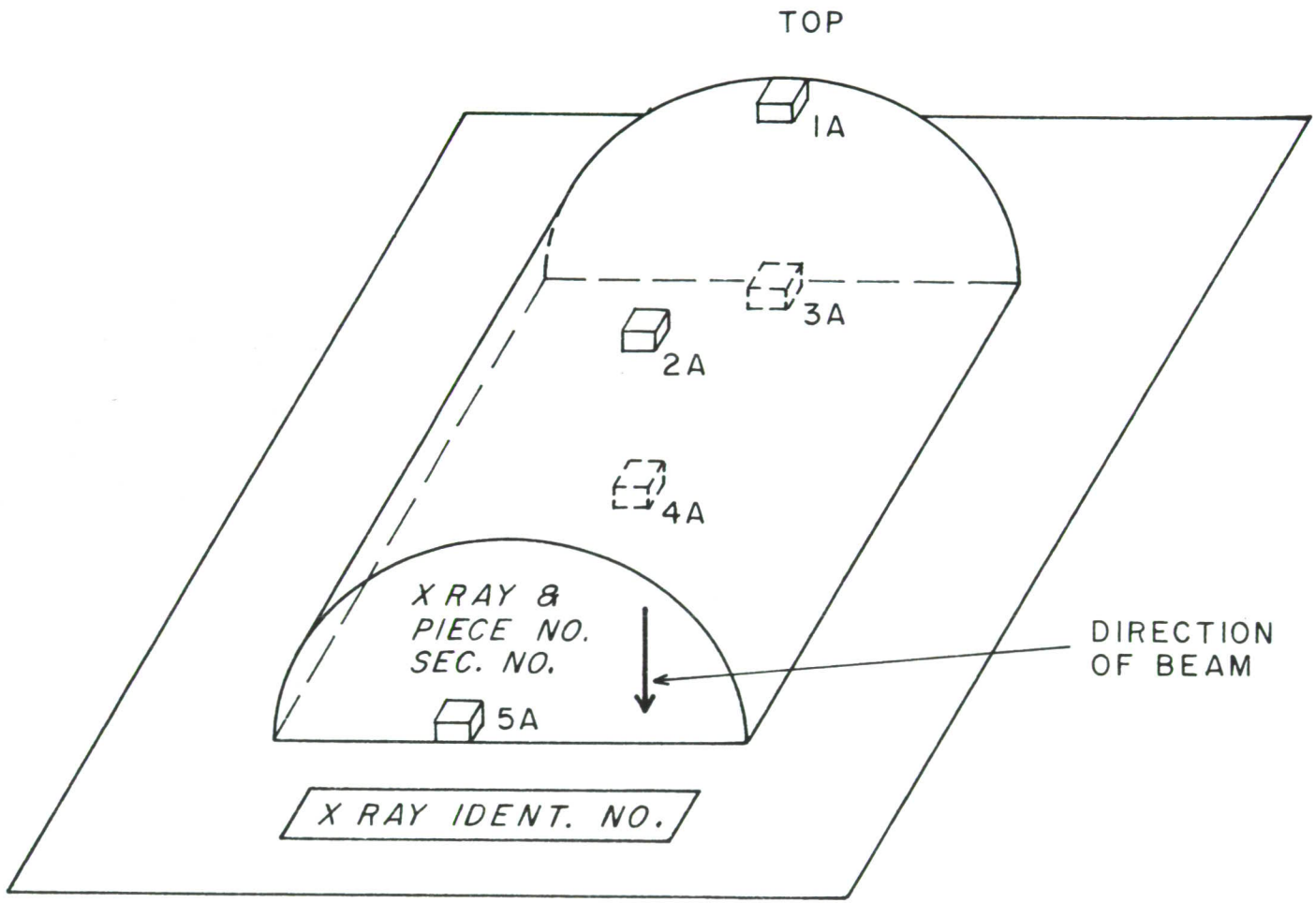


Fig. 7 - SECTION OF 7 IN. DIAMETER BY 6 1/2 IN. LONG BILLET FOR RADIOGRAPHIC EXAMINATION INDICATING LOCATIONS FROM WHICH SPECIMENS FOR MICROPROBE, X-RAY, AND METALLOGRAPHIC ANALYSES WERE OBTAINED

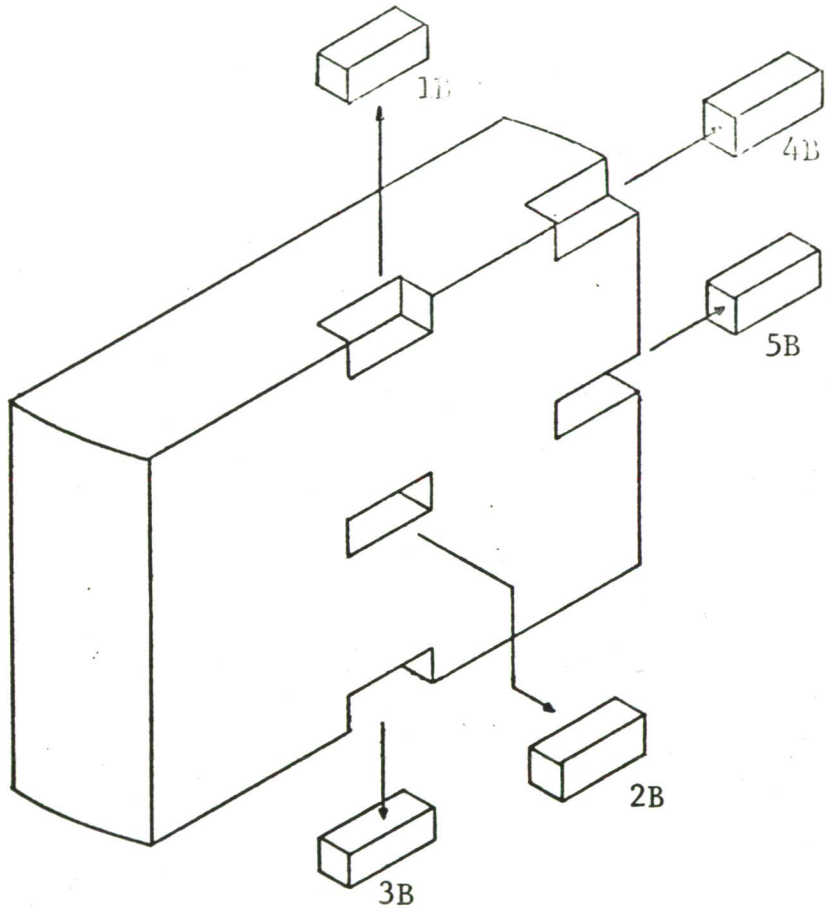


Fig. 8 - SECTION OF 14 IN. DIAMETER BY 7 IN. LONG BILLET FOR RADIOGRAPHIC EXAMINATION INDICATING LOCATIONS FROM WHICH SPECIMENS FOR MICROPROBE, X-RAY, AND METALLOGRAPHIC ANALYSES WERE OBTAINED

Table III

CHARACTERIZATION OF JTA GRAPHITE

FROM X-RAY ANALYSIS

Location (see Fig. 7)	Composition
1A	Graphite + ZrB_2 + βSiC
2A	Graphite + ZrB_2 + (Trace) βSiC
3A	Graphite + ZrB_2 + (Trace) βSiC
4A	Graphite + ZrB_2 + βSiC
5A	Graphite + ZrB_2 + βSiC

B. Spectrographic Analysis

Since x-ray analysis is effective only to about a 5% material content, other impurities and the formation of other carbides or silicides of less than 5% content were determined through a spectrographic analysis, Table IV. This work was performed by the Chicago Spectro-Service Laboratory, Inc.

The assumption that these impurities were volatilized even though the base material was not, is only true for Al, Fe, Ba, Cu, Co, Mn, and Ca, and probably is not true for Ti, Mo, V, and Cr. Thus the analysis holds for the lighter elements and those which do not form refractory carbides.

Examination of Table IV shows considerable impurity variation between billets for Al, Fe, Mo, V, Mn, and Cr. Since samples of the original powders from which these billets were formed have not as yet been made available, no analysis can be made for the source of these impurities. The maximum amount of impurities found was less than the 1.5% limit in the material specification.

C. Metallographic Analysis

Figures 9, 10, and 11 show the structure of the JTA graphite billets. The light areas are zirconium diboride, the dark gray areas are graphite, and the light gray areas are silicon carbide. In general, the graphite is uniformly distributed in the billets while the amount of zirconium diboride and silicon carbide varies. There appears to be a slight difference in the quantitiy of these materials and size of grains between the interior of the billets and the outside edges. The average size of the graphite grain appears to be 120 ± 30μ x 70 ± 15μ, the size of the zirconium diboride varies from about 100μ to less than 1μ, and the silicon carbide varies from 50μ to less than 1μ. Billets 7-F-12 and 7-F-14 are similar in structure in that material from the center of the billet shows random distribution of the various phases while the edge material appears to have a higher concentration of small grains of zirconium diboride with silicon carbide predominating at the graphite grain boundaries. Also, there appears to be somewhat less zirconium diboride in the edge material. An exception to the above generalization is the view from the top center of billet 7-F-14, which shows a structure similar to the edge material. Other work[4] in hot-pressing graphite-carbide composites has shown that low-melting phases will be squeezed out of the structure and diffused into the mold during hot-pressing and the remaining material will concentrate along the grain boundaries. The 7 in. diameter JTA graphite composite appears to be reacting in a similar manner. The 14 in. material exhibits a much greater homogeneity of

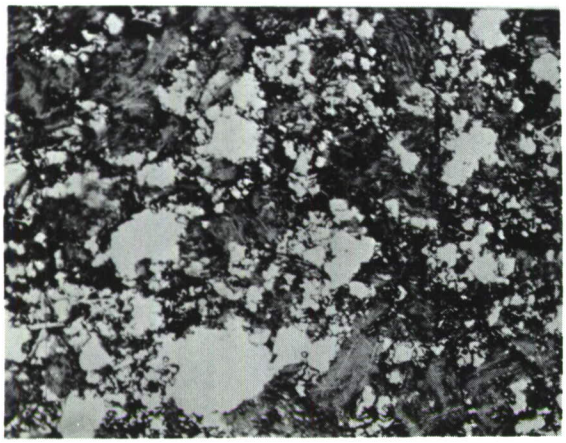
Table IV

SEMIQUANTITATIVE ANALYSES (WT%)
OF JTA GRAPHITE

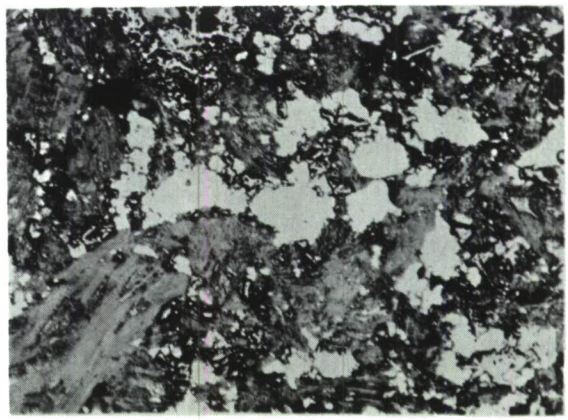
Sample No.	7-F-12	7-F-14	14-G-1
Zirconium	**	**	**
Boron	**	**	**
Silicon	**	**	**
Aluminum	0.05	0.2	0.2
Iron	0.2	0.08	0.05
Titanium	0.1	0.1	0.1
Molybdenum	0.1	<0.01*	<0.01*
Barium	0.03	0.03	0.03
Copper	0.02	0.03	0.03
Vanadium	0.02	0.005	0.005
Cobalt	0.01	0.01	0.01
Manganese	0.01	0.005	0.005
Chromium	0.01	0.001	0.002
Calcium	0.005	0.005	0.005
Magnesium	0.0002	0.0004	0.0002

*Trace

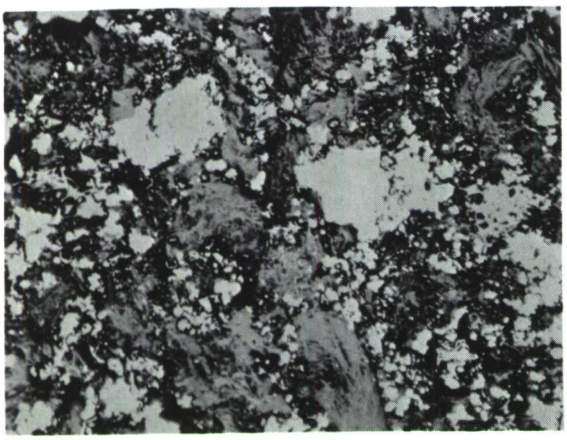
**The major portion of the base material present in these samples was not volatilized in the D.C. arc discharge; therefore, we cannot give any quantitative results for the principal elements. The figures shown are based on the assumption that the impurities were volatilized even though the base material was not.



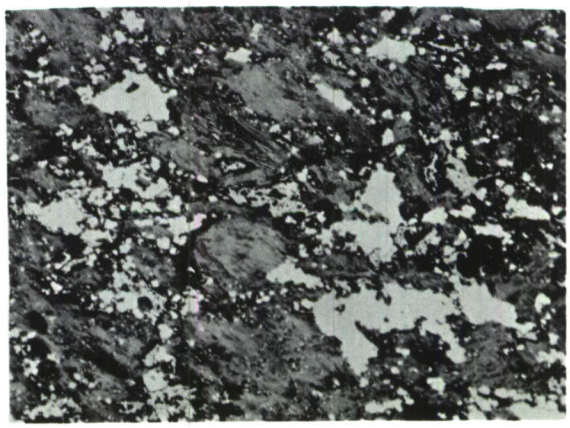
1A Location



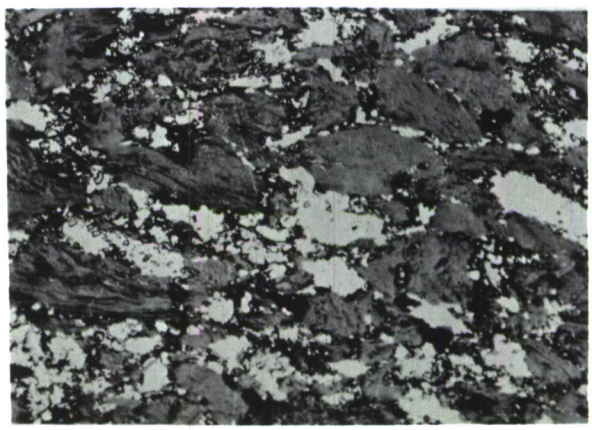
3A Location



2A Location



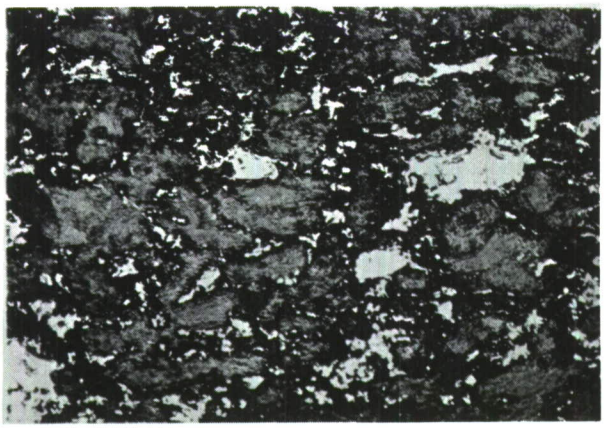
4A Location



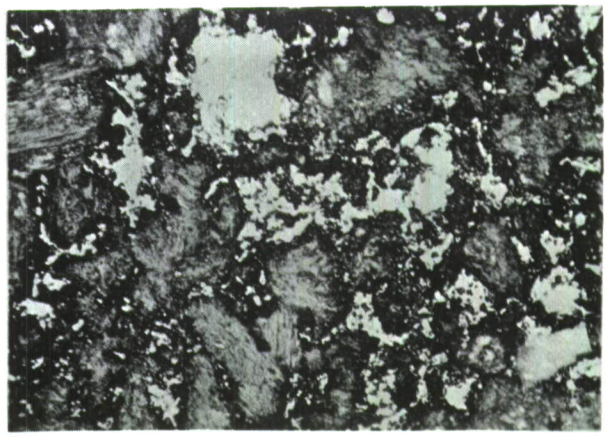
5A Location

50μ

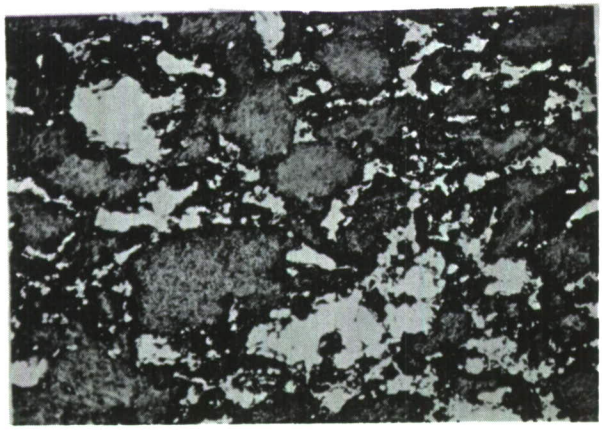
Fig. 9 - PHOTOMICROGRAPHS FROM JTA BILLET 7-F-12



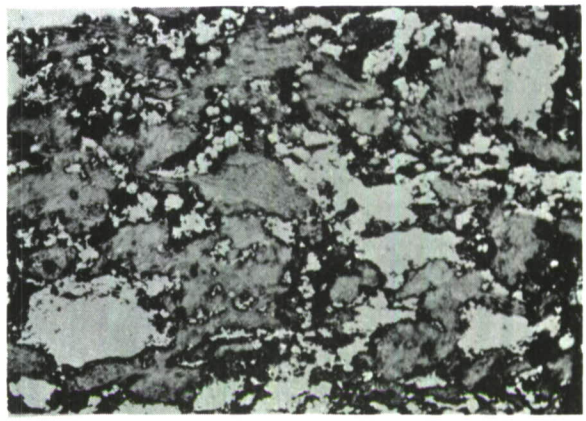
1A Location



3A Location

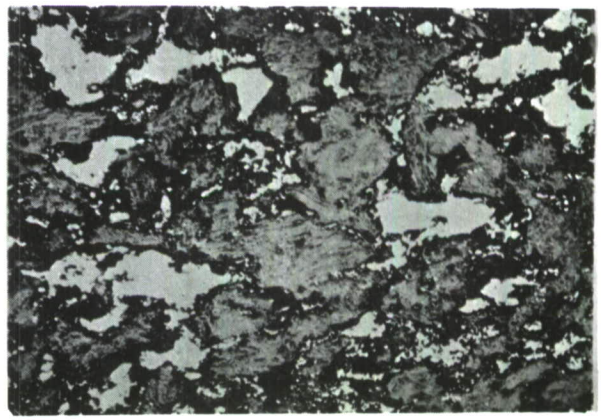


2A Location



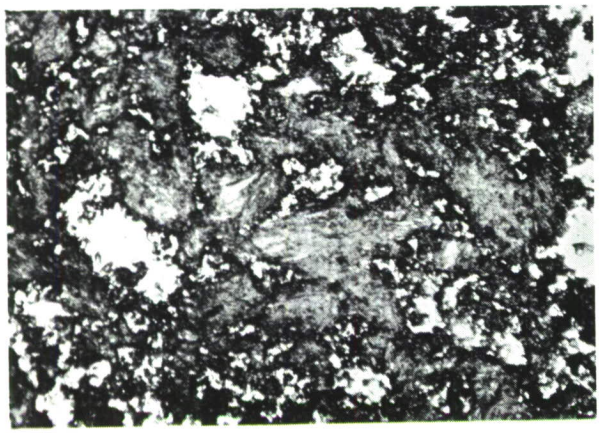
4A Location

50μ

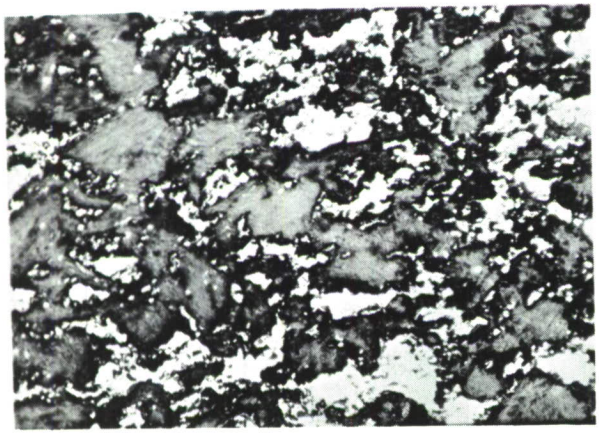


5A Location

Fig. 10 - PHOTOMICROGRAPHS FROM JTA BILLET 7-F-14

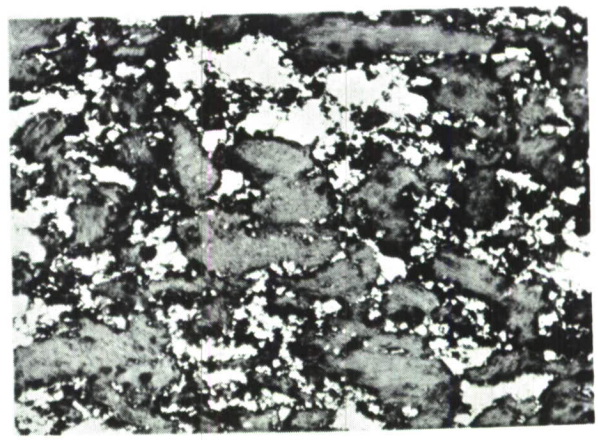


1A Location

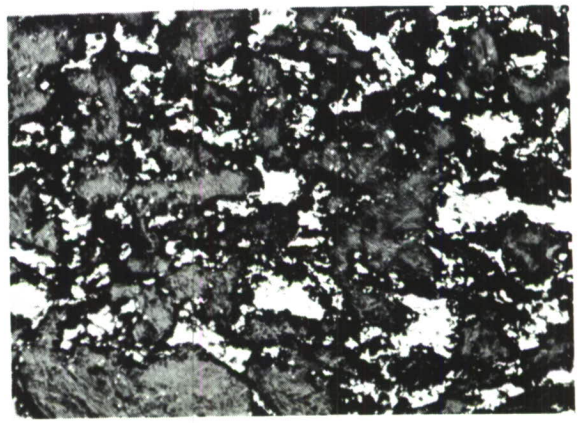


2A. Location

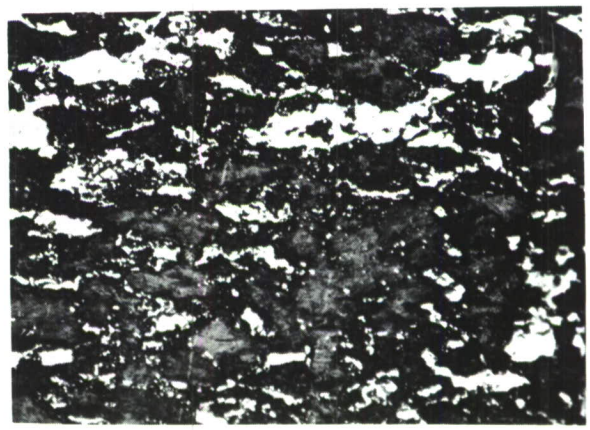
50μ



3A Location



4A Location



5A Location

Fig. 11 - PHOTOMICROGRAPHS FROM JTA BILLET 14-G-1

structure than the 7 in. material, although the trend to grain boundary segregation of small grains of second and third phase materials is still present. The billets are hot-pressed in the form of truncated cones and are machined to a cylindrical shape. Since the larger billet probably has more material cut away than the smaller billets, the evidence of edge segregation would be less for the larger billets than the smaller sizes. The effect of structural variability will be observed through a correlation with mechanical strength.

Close examination of the samples under the microscope at about 1000 magnification shows what appear to be microcracks in the zirconium diboride. Once located, they can also be seen in the lower magnification (320X) photomicrographs shown in the report. No extensive analysis of these microcracks has as yet been made. However, they have also been seen by L. Marcus[5] in a study at Bell Aerosystems Company.

D. Electron Microprobe Analysis

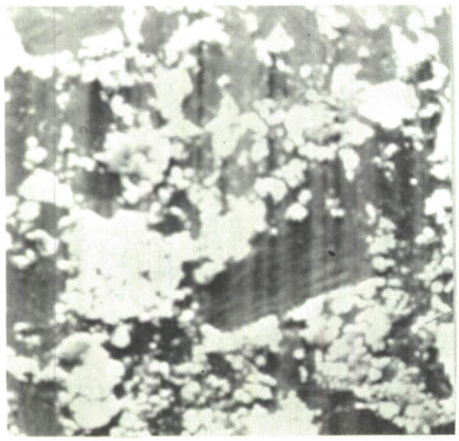
Figure 12 provides the results of microprobe analysis taken at position 3A of billet 7-F-12. The equipment at ETKI can detect an amount of material above 5 wt% with an error of + 5 wt%. Penetration depth is 5 wt% for C, Si, and B, and 1 wt% for Zr.

While the microprobe confirms the x-ray work that graphite, zirconium, boron, and silicon are present, the results of the analysis are inconclusive as to the amounts and location of these elements with respect to x-ray analysis and the literature.

Theoretically, in the zirconium-rich area there should be 81 wt% Zr and 19 wt% B and in the silicon-rich area there should be 70 wt% Si and 30 wt% C. Actual analysis (Fig. 10) does not conform to the theoretical results. In the carbon-rich area less than 5 wt% Si, Zr, and B are present. In the zirconium-rich area, 46 wt% Zr, 22 wt% B, and less than 5 wt% C and 1 wt% Si were found; in the silicon-rich area, 58 wt% Si and less than 5 wt% C, Zr, and B were found.

The conclusion drawn from the above data is that free silicon and little SiC are present, and this is very unlikely. This phase of the program is still under study. The microprobe sees only a very small area; when more data are available, it should be possible to make better correlations with the other available information.

If the literature values for component materials are assumed to be correct, the actual weight per cent of the three major components of JTA graphite should be 43 wt% ZrB_2, 13 wt% SiC, and 44 wt% C.

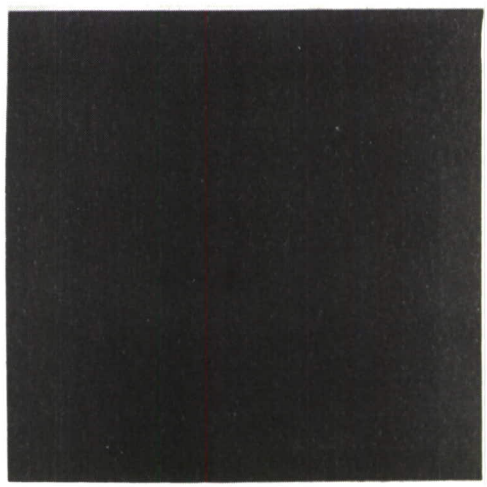


BEAM SCAN

CARBON

ZIRCONIUM

SILICON



BORON

Fig. 12 - MICROPROBE ANALYSIS OF BILLET 7-F-12, SECTION 3A

E. Density

Specification requriements are that the bulk density of the material to be delivered to IITRI be greater than 3.00 g/cc. Bulk density measurements confirm the uniformity shown by the radiographic and metallographic analyses. Random measurements of 20 samples from each billet show very little variation through the centers of each billet; 7-F-12 averages 3.063 g/cc, 7-F-14 averages 3.077 g/cc, and 14-G-1 averages 3.091 g/cc. See Table V for complete data.

IV. MECHANICAL PROPERTIES

A series of preliminary studies were made to: (1) determine reasonable sample sizes and shapes, and (2) determine the effect of oxidation on strength, under various pressures and times of exposure. The size effect experiments were performed to show if a specimen cross-section with a minimum dimension of 1/4 in. will have strengths which are typical of bulk material.

A. Size Effect Study

Experiments were performed at room temperature. The data were fitted to a statistical size effect analysis which provided the necessary information on reliability of results from specimens with three different cross-sectional areas.

1. Flexure Tests

Table VI lists the specimen size and the total number tested for this phase of the program. All flexure testing was done using a 4-point loading system with the load points at the end of the specimen and at the third points. The distance between the points was maintained at a 10:1 ratio with regard to the depth of the cross section. Surface finishes for these specimens averaged 20-50 rms for both grain orientations. In order to procure as much data as possible from these tests,sonic modulus data were obtained for all specimens tested. In addition, the deflection of each specimen under load was measured (Fig. 13) and the static Young's elastic modulus calculated. Eighteen of the 1/2 x 1/2 x 6 in. specimens were instrumented with strain gages, and both the longitudinal and lateral strains recorded under load for "with" and "across" grain orientations. Young's elastic modulus and Poisson's ratio can be calculated from this information. The data for each property measurement were correlated with the other properties for each specimen. The bending fixture is rigidly fastened to the moving head, and the specimen is supported on knife edges which are free to rotate in two directions; Fig. 14 shows this test setup.

Table V

SAMPLE DENSITIES (g/cc) THROUGH CENTER OF JTA GRAPHITE BILLETS

14-G-1	7-F-14	7-F-12
3.093	3.040	3.082
3.065	3.071	3.112
3.067	3.118	3.082
3.103	3.052	3.082
3.102	3.063	3.082
3.073	3.048	3.107
3.116	3.053	3.112
3.088	3.042	3.076
3.141	3.103	3.082
3.114	3.085	3.132
3.074	3.101	2.982
3.067	3.072	3.032
3.116	3.103	3.047
3.083	3.083	3.077
3.086	3.115	2.954
3.072	3.085	2.984
3.091	3.057	3.041
3.065	3.068	3.087
3.117	3.095	3.079
3.088	3.082	3.090
3.091 Avg	3.077	3.065
±0.021 Std. Dev.	±0.023	±0.049

Table VI

DETERMINATION OF SIZE EFFECT OF JTA GRAPHITE SPECIMENS AT ROOM TEMPERATURE

(Flexure Testing)

Specimen Length, in.	Cross-Section Dimensions, in.	No. of Specimens	
		With Grain	Across Grain
1	1/16 x 1/16	30	30
3	1/4 x 1/4	30	30
6	1/2 x 1/2	30	30

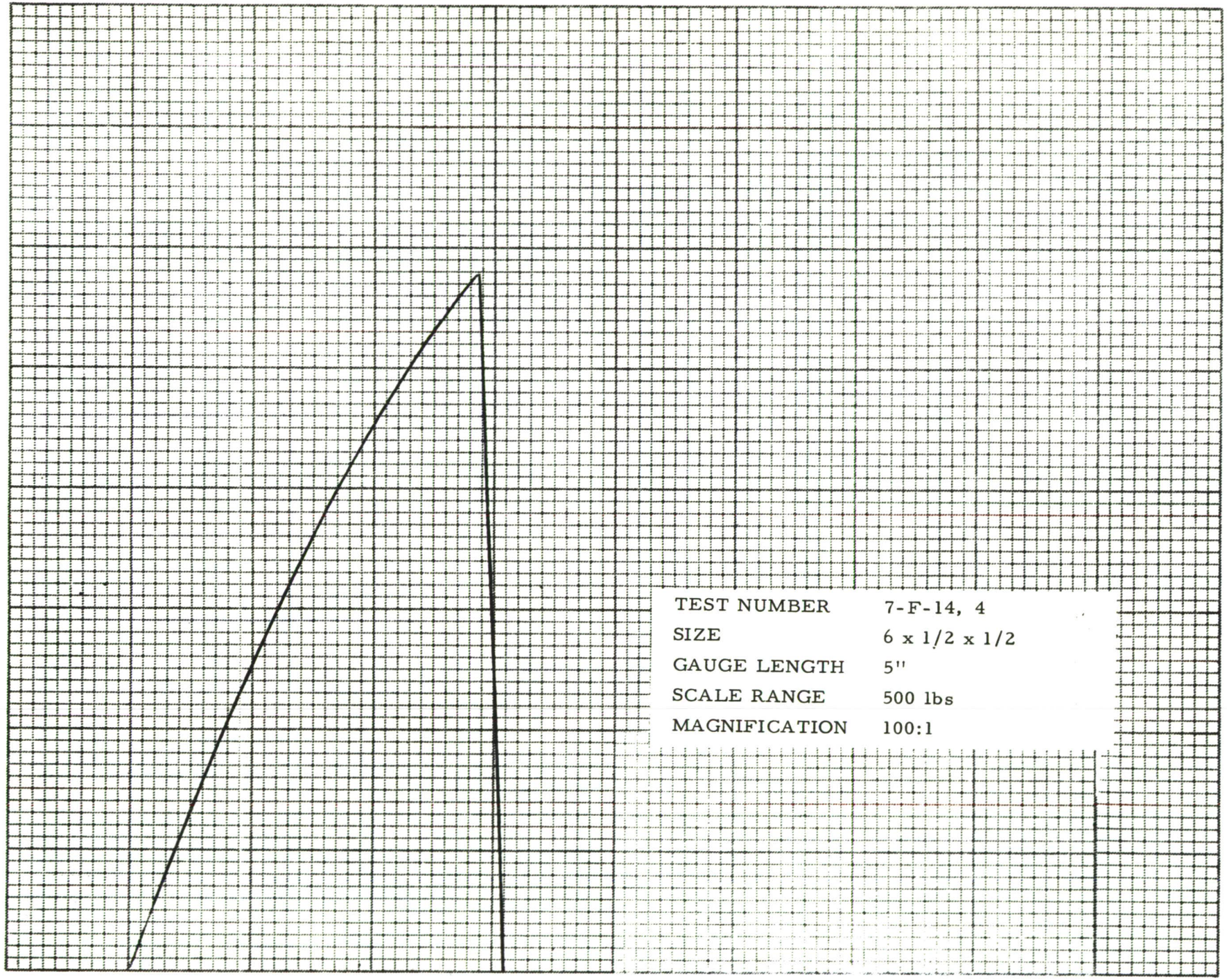


Fig. 13 - LOAD-DEFLECTION CURVE

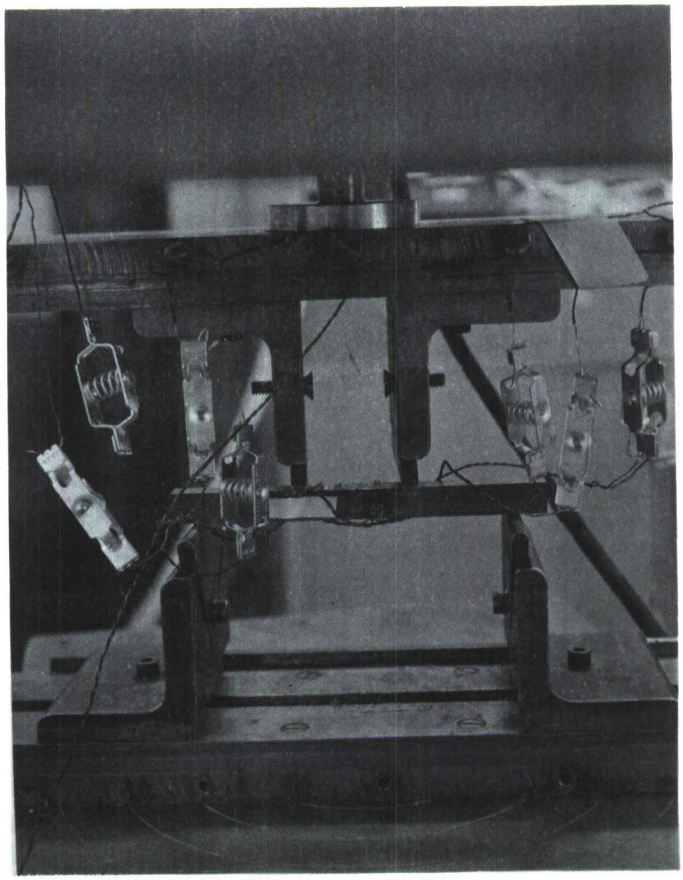


Fig. 14 - TEST SETUP FOR OBTAINING POISSON'S RATIO WITH SR-4 STRAIN GAGES

Tables VII, VIII, and IX list the results obtained during the size effect experiments. In all cases, there is an apparent increase in strength with decreasing cross section except for the 1/16 x 1/16 x 1 in. "across" grain specimens. These either exhibit about the same strength, or show a loss in strength compared to the 1/4 x 1/4 x 3 in. or the 1/2 x 1/2 x 6 in. specimens. In all cases, the 1/16 x 1/16 x 1 in. specimen exhibited a large increase in variability. As previously shown, the graphite grain size averages about 120 $\pm$ 30μ (0.00485 in.) long and 70 $\pm$ 15μ (o.0027 in.) wide. The 1/16 in. cross section reduces to 0.0625 in., which is approximately 13 times the average grain size. Thus, these experiments apparently verify the rule-of-thumb relationship that the depth of the beam must be greater than ten times the largest dimension of the grain in the cross section. Since the grain dimensions given are averages, it is easy to account for the large variability exhibited by the "across" grain specimens of 1/16 in. cross-sectional area. Table X and Fig. 15 provide a summary analysis of the size effect data.

2. Tension Tests

Tensile strength is being measured using direct techniques. Preliminary experiments were made to determine whether the pin-type specimen (Fig. 16) offers any advantages over a collet grip specimen (Fig. 17). The latter test grips (Fig. 18) were developed on another IITRI program. Since JTA is a high-cost material, the preliminary experiments were made using a less costly graphite, POCO EP 1924. Table XI lists data obtained from these experiments. The literature strength values for the tensile strength of EP 1924 is 6000 psi. These experiments, while limited in scope, indicate that the collet specimens appear to provide more reliable data than the pin-specimen. On the basis of these tests the collet specimen was chosen for use in this program.

In order to study the critical size range and eliminate the problem due to fabricating small 1/16 in. diameter gage sections, cross-section diameters of 1/8, 3/16 and 1/4 in. were studied. Figure 19 is a plot of these data. The "with" grain plot exhibits a certain amount of inconsistency,and it is conceivable that the dotted line is a better average plot of their values than the solid curve line because of the small population groups used. The conclusion to be drawn from these data is that a 1/4 in. cross section will provide an accurate representation of the JTA graphite strength in tension as well as flexure.

Table VII

EXPERIMENTAL DATA ON SIZE EFFECT FOR BILLET 7-F-14

Specimen Cross-Section,* in.	Flexural Strength Avg, psi	Flexural Strength Coef. of Error, %	Tangent Modulus Avg, psi	Tangent Modulus Coef. of Error, %	Sonic Modulus Avg, psi	Sonic Modulus Coef. of Error, %
			With Grain			
6 x 1/2 x 1/2	14,972	9.89	8.36	10.10	18.00	5.94
3 x 1/4 x 1/4	18,352	6.73	10.42	11.13	19.36	1.86
1 x 1/16 x 1/16	20,428	7.49	---	---	4.20	9.29
			Across Grain			
6 x 1/2 x 1/2	7,100	7.91	3.26	10.61	9.78	5.62
3 x 1/4 x 1/4	9,306	3.88	4.56	10.09	13.57	3.54
1 x 1/16 x 1/16	8,704	16.29	---	---	3.65	3.56

*10 specimens of each cross-section were tested.

Table VIII

EXPERIMENTAL DATA ON SIZE EFFECT FOR BILLET 7-F-12

Specimen Cross-Section,* in.	Flexural Strength Avg, psi	Flexural Strength Coef. of Error, %	Tangent Modulus Avg, psi	Tangent Modulus Coef. of Error, %	Sonic Modulus Avg, psi	Sonic Modulus Coef. of Error, %
			With Grain			
6 x 1/2 x 1/2	14,820	22.3	9.4	21.5	14.6	9.5
3 x 1/4 x 1/4	17,320	21.2	13.0	21.3	17.1	2.5
1 x 1/16 x 1/16	23,220	19.0	7.2	29.1	5.1	21.8
			Across Grain			
6 x 1/2 x 1/2	8,640	8.1	4.2	8.3	10.5	8.5
3 x 1/4 x 1/4	9,370	11.5	4.0	44.6	6.8	3.9
1 x 1/16 x 1/16	7,580	43.5	4.6	17.7	5.25	15.7

*10 specimens of each cross-section were tested.

Table IX

EXPERIMENTAL DATA ON SIZE EFFECT FOR BILLET 14-G-1

Specimen Cross-Section,* in.	Flexural Strength Avg, psi	Flexural Strength Coef. of Error, %	Tangent Modulus Avg, psi	Tangent Modulus Coef. of Error, %	Sonic Modulus Avg, psi	Sonic Modulus Coef. of Error, %
			With Grain			
6 x 1/2 x 1/2	15,818	15.99	9.32	14.37	15.30	9.67
3 x 1/4 x 1/4	17,880	13.23	9.83	13.22	18.31	4.15
1 x 1/16 x 1/16	24,190	11.58	---	---	4.58	10.26
			Across Grain			
6 x 1/2 x 1/2	8,800	5.32	4.13	8.29	10.50	7.00
3 x 1/4 x 1/4	8,160	2.92	4.99	10.62	15.47	3.04
1 x 1/16 x 1/16	9,045	22.54	---	---	3.72	7.80

*10 specimens of each cross-section were tested.

Table X

COMBINED SIZE EFFECT DATA

Specimen Cross-Section,* in.	Flexural Strength Avg, psi	Flexural Strength Coef. of Error, %	Tangent Modulus Avg, psi	Tangent Modulus Coef. of Error, %	Sonic Modulus Avg, psi	Sonic Modulus Coef. of Error, %
			With Grain			
6 x 1/2 x 1/2	15,203	14.37	9.26	16.63	15.97	12.52
3 x 1/4 x 1/4	17,850	14.87	11.08	21.14	18.26	5.46
1 x 1/16 x 1/16	22,613	15.59	---	---	4.63	17.71
			Across Grain			
6 x 1/2 x 1/2	8,180	11.77	3.86	14.22	10.26	7.89
3 x 1/4 x 1/4	8,945	9.73	4.52	26.06	11.95	30.79
1 x 1/16 x 1/16	8,443	29.18	---	---	4.21	21.31

*Total of 30 specimens for each cross-section were tested.

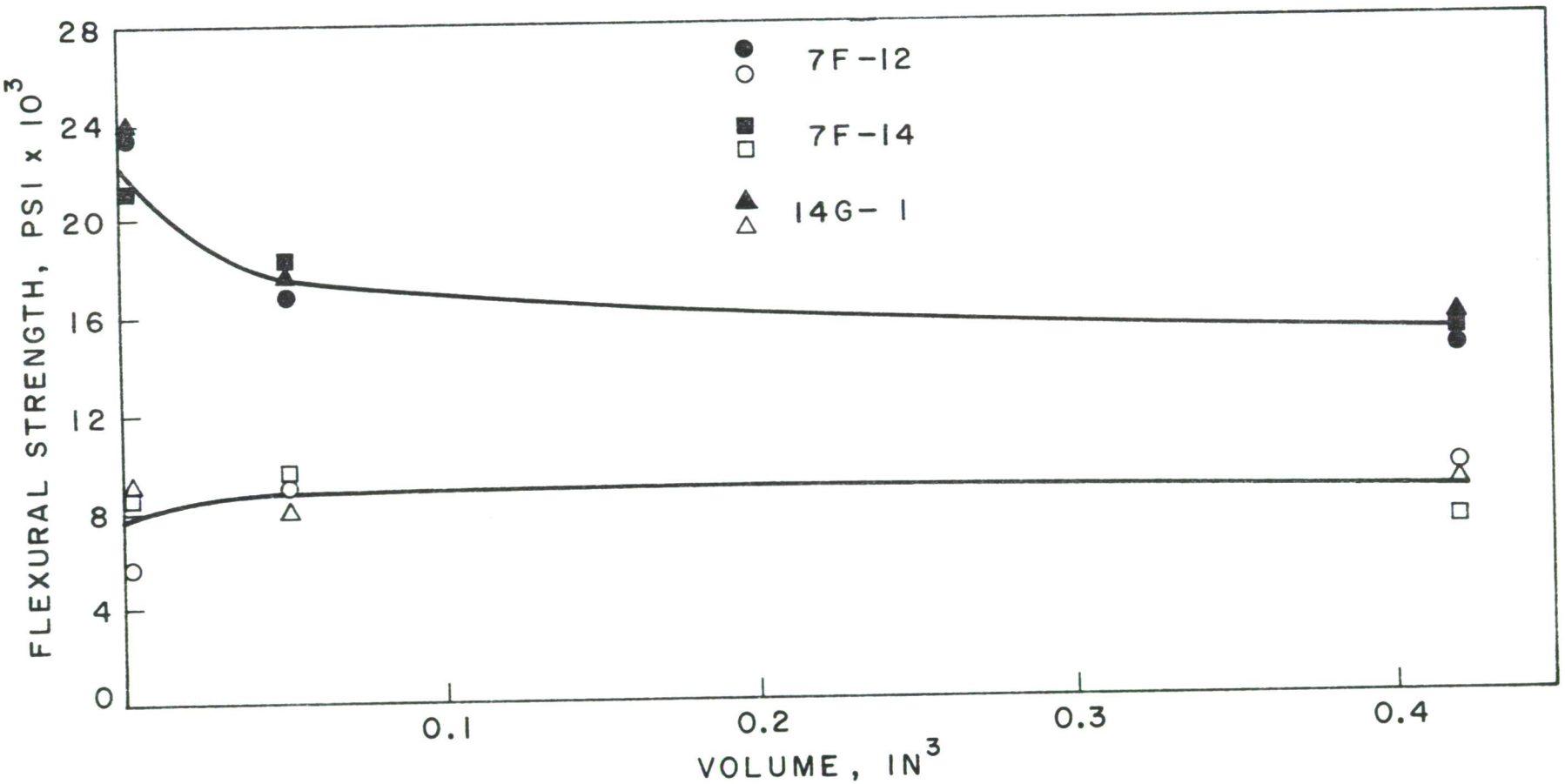


Fig. 15 - SIZE EFFECT ON FLEXURAL STRENGTH

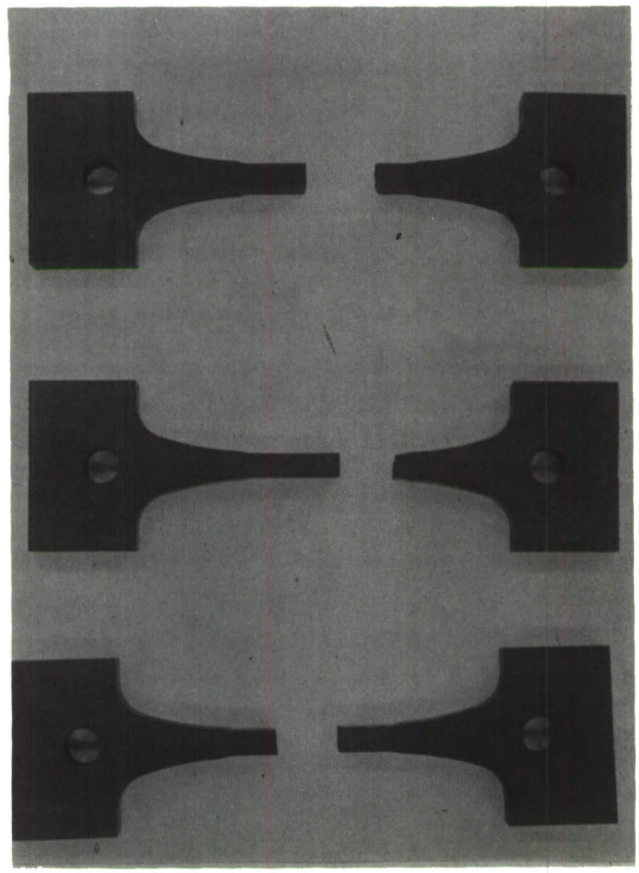


Fig. 16 - PIN-TYPE TENSION SPECIMEN
SHOWN WITH TYPICAL FRACTURES

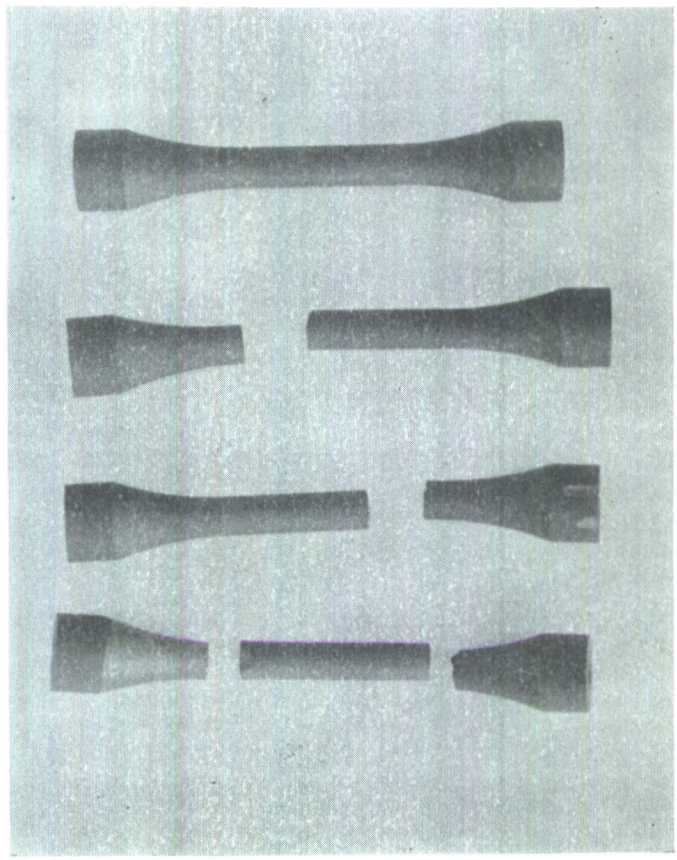


Fig. 17 - COLLET TENSION SPECIMEN
SHOWN WITH TYPICAL FRACTURES

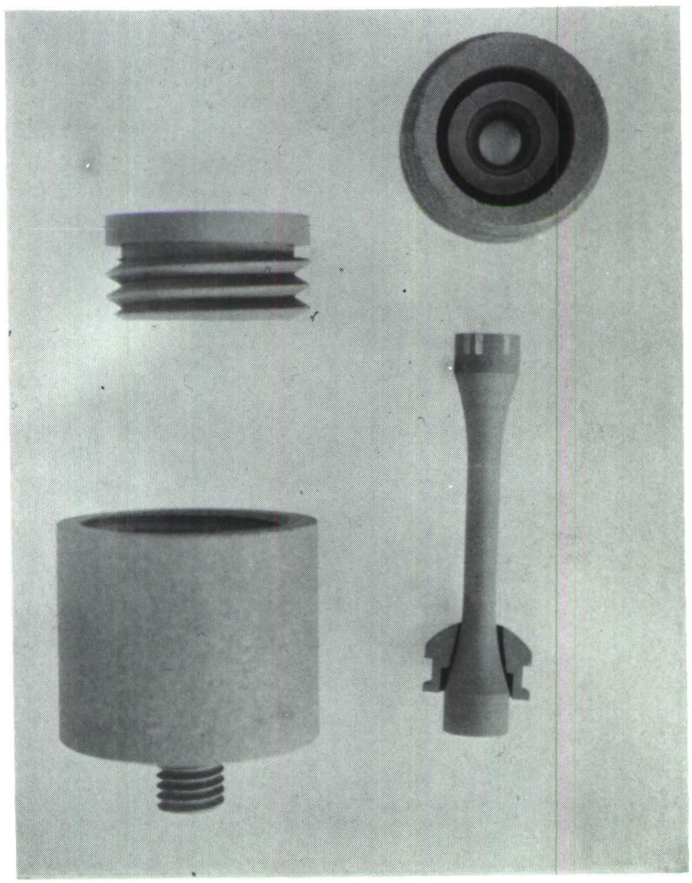


Fig. 18 - TENSION GRIPS DEVELOPED AT IITRI
TO BE USED WITH COLLET SAMPLES

Table XI

TENSILE STRENGTH OF POCO GRAPHITE EP 1924

Type of Tension Specimen	No. of Tests	Tensile Strength, psi	Coefficient of Variation, %
Pin-type	5	6480	19.5
Collet type	7	6140	14.2

Table XII

ROOM-TEMPERATURE ELASTIC PROPERTIES OF JTA GRAPHITE

Billet No.	Modulus in Flexure 10^6 psi		Poisson's Ratio in Flexure	
	Tension	Compression	Tension	Compression
		With Grain		
7-F-12	5.535	6.268	.233* .082**	.309* .125*
7-F-14	8.100	9.125	.252 .079	.321 .135
		Across Grain		
7-F-12	3.139	3.493	0.119	0.145
7-F-14	3.140	4.074	0.093	0.151
14-G-1	4.397	4.279	0.110	0.116

* Poisson's ratio determined in A direction.
** Poisson's ratio determined in C direction.

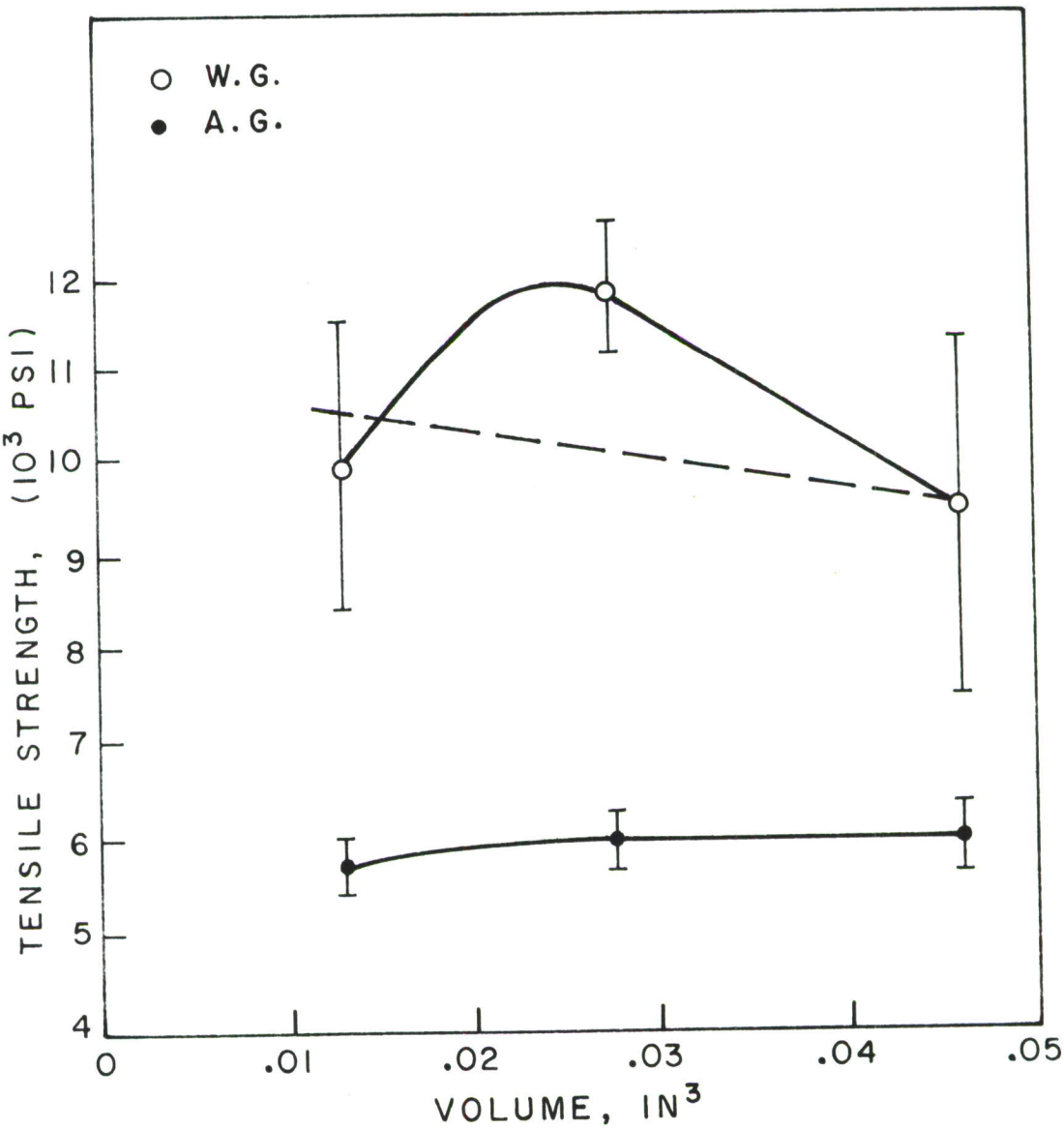


Fig. 19 - SIZE EFFECT ON TENSILE STRENGTH.

B. Elastic Properties

In order to obtain as much data as possible from the size effect experiments, SR-4 electric resistance gages were fastened to 5 specimens from each billet to determine Poisson's ratio. Two gages were applied to each of the four longitudinal faces; one measured the longitudinal strain and one the cross strain. Thus the elastic modulus and Poisson's ratio were measured for two orientations of "with" and "across" grain relationship. The test setup is shown in Fig. 14. Table XII presents the results of these experiments. It is obvious from the data that there are two Poisson's ratios which have to be considered, depending on the microstructure--one for the "A" direction and one for the "C" direction orientation; the first represents the broad face of the graphite crystallite and the second the edge of the crystallite. The "with" grain orientation will exhibit Poisson's ratio for both axes depending on how the sample is oriented for measurement.

In addition, the elastic properties in compression are higher, as expected, than that exhibited in tension. Thus, in order to obtain a true flexure strength the data must be corrected for the true position of the neutral axis of the bars.

Sonic elastic modulus measurements are generally higher than the static elastic modulus obtained from deflection or direct strain measurements. In order to verify the sonic modulus reported on this program, a "with" grain specimen of JTA graphite was obtained from Carbon Products Division of Union Carbide Corporation, and the values obtained using their equipment were compared to those obtained using IITRI equipment. Carbon Products obtained a value of $12.89 \pm 0.5 \times 10^6$ psi, and IITRI measured the sonic modulus as $13.66 \pm 0.4 \times 10^6$ psi--a difference of 6%. Considering that different equipment and techniques were used, the agreement between measurements is good and provides confidence in the reliability of the sonic measurements.

C. Evaluation of Flexure Strength Test Data

The failure distribution for the 1/4 in. cross section flexure strength data is plotted in Figs. 20 and 21. An analysis of variance shows that all the strength data come from the same population (see Appendix) at the 95% confidence level. However, examination of the curves reveals several factors of additional interest.

A rank sum test[6] comparing the three sample populations reveals that while the "with" grain orientation data can be considered from the same material population, the "across" grain populations are not the same. However, the narrow failure distributions of the "across" grain population have average strengths

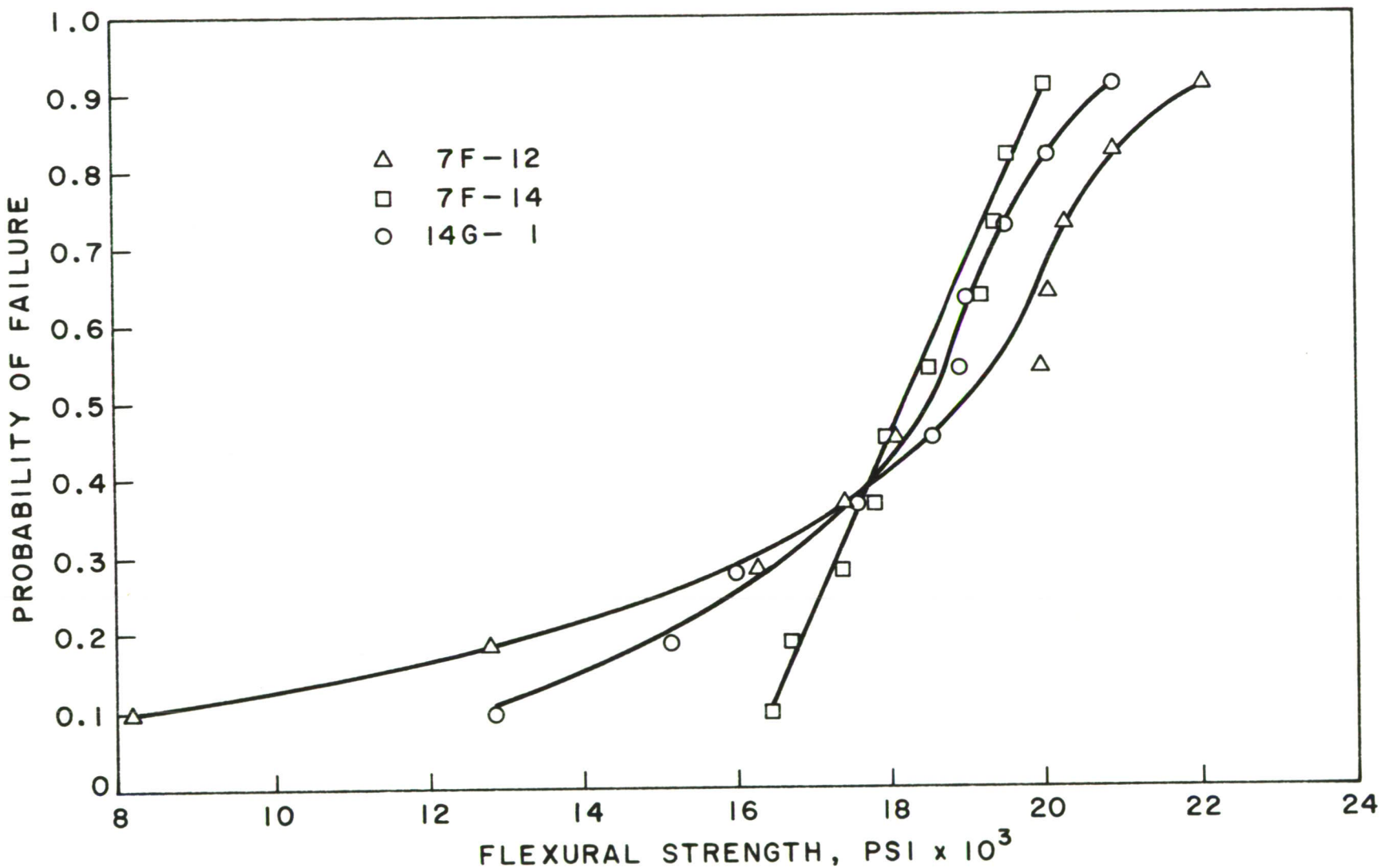


Fig. 20 - FLEXURAL STRENGTH DISTRIBUTION CURVES, "WITH" GRAIN ORIENTATION

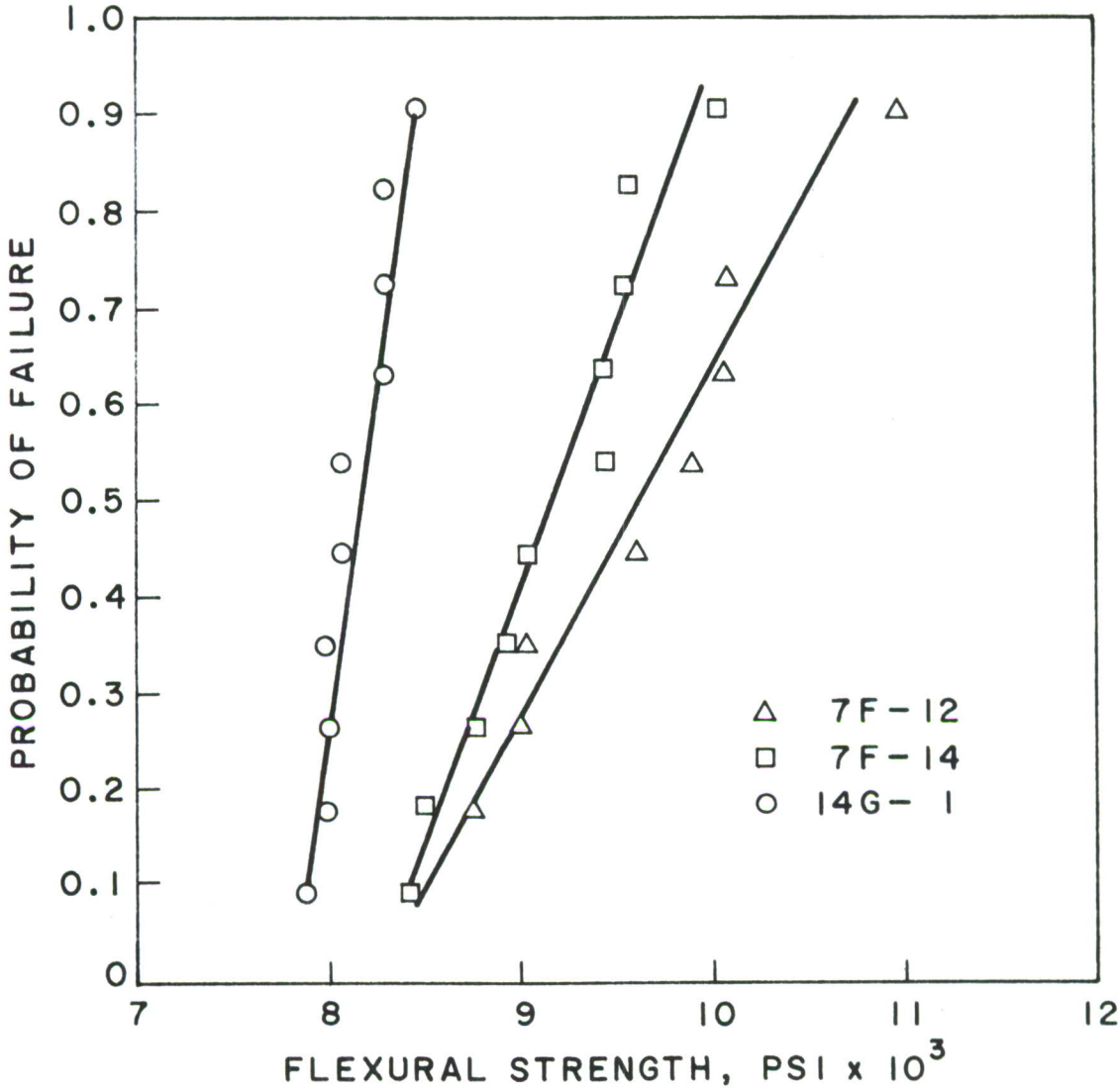


Fig. 21 - FLEXURAL STRENGTH DISTRIBUTION CURVES, "ACROSS" GRAIN

which are very nearly the same. Thus, while the two statistical analyses are partially contradictory, the test data of the three billets are considered as part of the same data population and the strengths of these billets are the same.

The failure modes of the "with" grain and "across" grain orientation appear to be different. The probability-of-fracture curves for "with" grain orientation are typical for brittle materials, in that they are S-shaped while the "across" grains are straight lines. The S-curves for brittle materials normally have a skew, bell-shaped distribution with a wide strength variation biased in the direction of low strength. The straight lines of the "across" grain plots are indicative of a square failure distribution curve of small variability.

If data from a previous program are added for analysis, a similar trend is evident. The anisotropy due to average crystallite orientation not only affects the strength, elastic, and thermal properties, but also affects the failure mode for JTA. A review of the available literature at IITRI has failed to reveal a similar analysis for any of the graphite materials. It may be that all graphites show this phenomenon. This behavior can probably be traced to the different bonding mechanisms of the graphite crystals, weak van der Waals forces for the average "across" grain orientation, and much stronger ionic bonding for the "with" grain orientation.

Combined failure strength curves are shown in Figs. 22 and 23. There is one data point for the "across" grain plot which is out of line with the rest of the data. A statistical analysis indicates that this point can be dropped as nonrepresentative of the material strength. It is conceivable that damage was introduced into this specimen during cutting. A similar analysis for the "with" grain specimen does not show these extraneous data points.

From a design point of view, the "across" grain data appear to be deterministic while the "with" grain data exhibit "statistical" behavior similar to oxide ceramics. As the rest of the mechanical property results become available, it will be interesting to observe if the data continue to group in the manner exhibited by the flexure data.

D. Oxidation Effects

JTA graphite was developed for use at high temperature under oxidizing conditions. Mechanical data obtained by standard techniques of testing in inert atmospheres will be correlated with those performed in air. First, a series of experiments are being conducted to determine the effect of oxidation on the strength of JTA. Flexure tests are being employed similar to the size effect experiments.

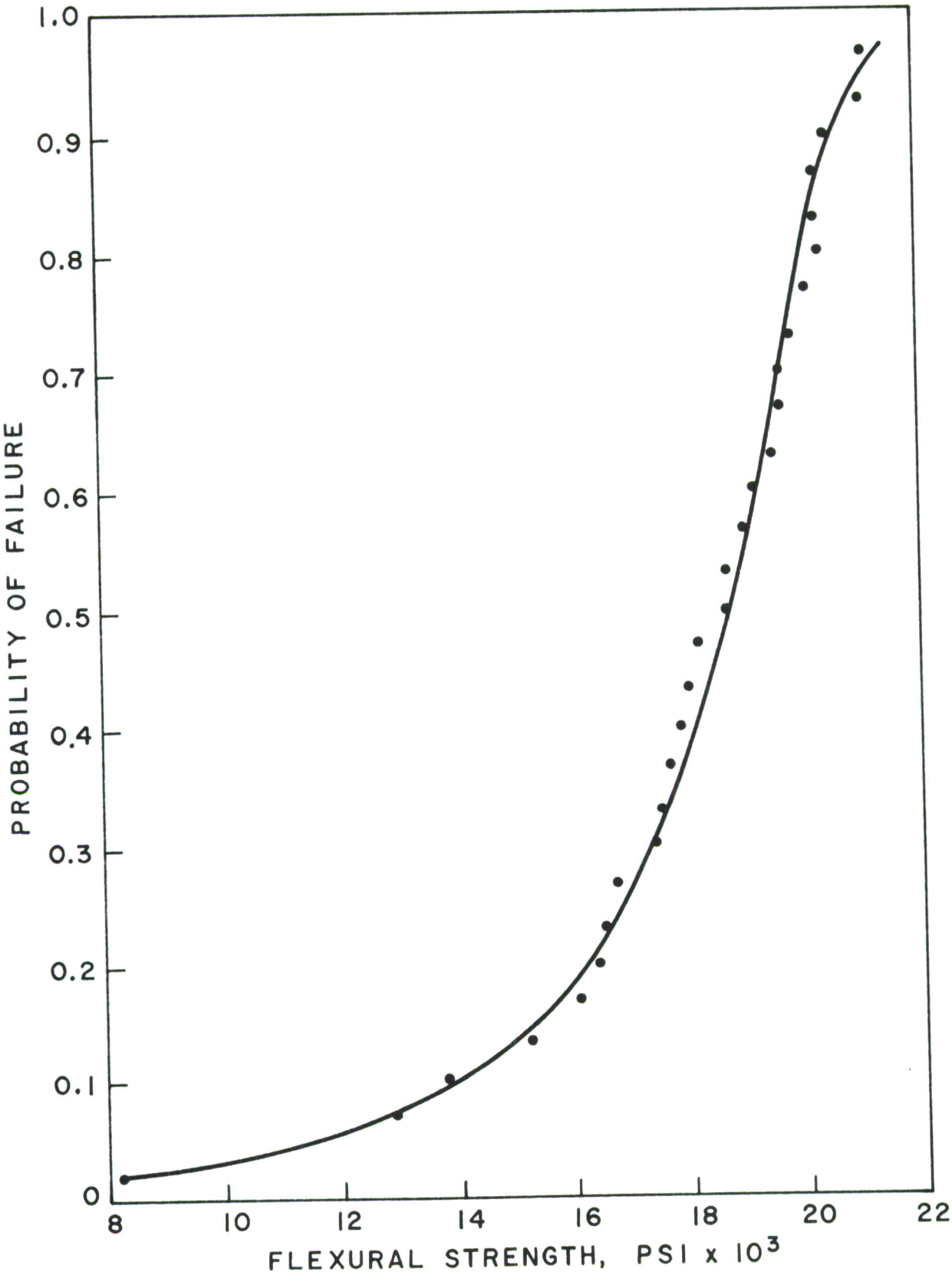


Fig. 22 - COMBINED FLEXURAL STRENGTH DISTRIBUTION CURVE, WITH GRAIN

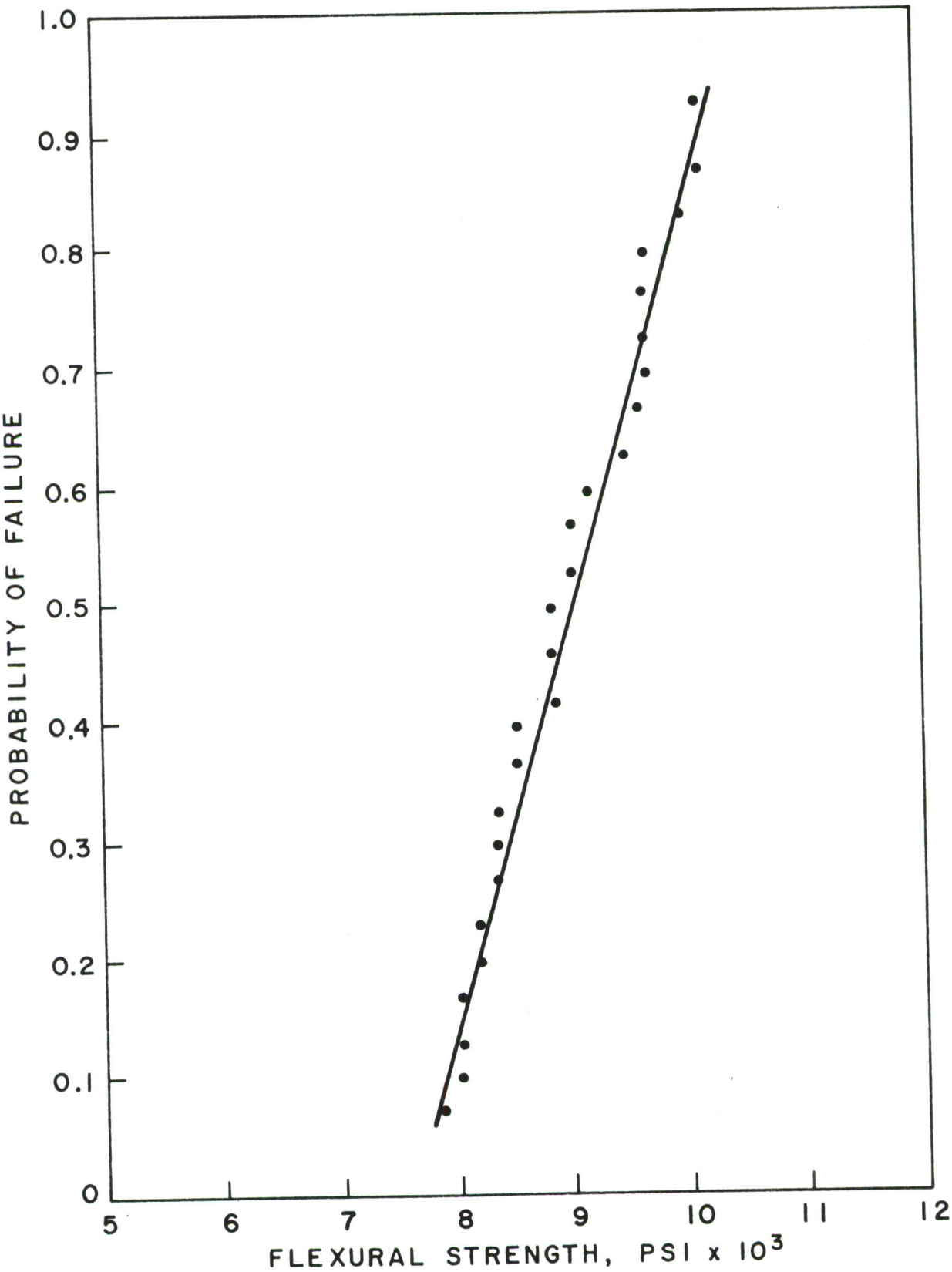


Fig. 23 - COMBINED FLEXURAL STRENGTH DISTRIBUTION CURVE, ACROSS GRAIN.

Specimens have been exposed to three time periods, at temperatures of 1000°, 1750°, and 2000°C for the following pressures: 1 x 10^{-5} Torr, 1 x 10^{-1} Torr and atmospheric. Three specimens are tested for each condition of air pressure and temperature.

The data from these experiments will be analyzed with flexural data obtained from tests in an inert atmosphere. After analysis, a similar series of tests will be conducted for tension and compressive strengths under oxidizing conditions (see Table XIII), and the data will be compared to results from inert atmosphere tests. These tests should enable the translation of mechanical property data taken in the inert environment to that from the oxidizing conditions; the depth or amount of oxidation, the nature of the oxidized material, the rate at which oxidation progressed, and the effect on strength will be determined.

Initial oxidation experiments have been initiated. A series of specimens have been exposed to static air at 1000°C, 10^{-5} Torr pressure and atmospheric pressure, and at 2000°C and 10^{-5} Torr pressure. Table XIV lists the strength obtained from room-temperature flexure tests after exposure. The material exposed at 1000°C in static air lost about 20% of its "with" grain strength in the first 20 minutes and retained the remainder through the next 40 minutes.

The "across" grain showed similar behavior; however, the strength loss was somewhat less, about 15%. These values are only tentative since more specimens must be evaluated before any final conclusions can be drawn.

The material tested at 2000°C in a vacuum exhibited little or no strength change: 5% for the "across" and 2% for the "with" grain orientation. This was expected since there was little or no air to effect oxidation changes.

Figures 24, 25, and 26 show weight changes that occurred during these experiments. The material exposed to static air at atmospheric pressure and 1000°C lost weight during the first 20 minutes, gained it back in the second 20 minutes, and then began to lose it again during the last 20 minutes. The curve for weight change at 1000°C at 10^{-5} Torr atmospheric pressure of static air is similar, except that the weight loss is about one order of magnitude less for the same time interval.

The material tested in vacuum at 2000°C only indicates loss of volatiles due to heating which should taper off after a certain amount of exposure. Under highly oxidizing conditions, both the volatiles and graphite are being lost, constituting a

Table XIII

OXIDATION TESTS OF JTA GRAPHITE

Property	Crystal Orientation	No. of Specimens at Temp. (°C) 1000	1750	2000
Tension	With grain	10	10	10
	Against grain	10	10	10
Flexure	With grain	10	10	10
	Against grain	10	10	10
Compression	With grain	10	10	10
	Against grain	10	10	10

Table XIV

OXIDATION EXPERIMENTS ON JTA 7-F-12 BILLET
(1/4 x 1/4 x 3 in. flexural specimens)

Grain Orientation	Avg Strength (at RT), psi	Time of Exposure, min	Flexure Strength, psi	Strength Change, %
		Static Air at 1000°C		
Across	8,945	20	8,060	-9.9
		40	6,400	-28.2
		60	7,040	-21.7
With	17,850	20	13,950	-21.8
		40	16,900	-5.3
		60	14,450	-19.1
		Static Air at 10^{-5} Torr and 2000°C		
Across	8,945	20	8,830	-1.3
		40	7,420	-17.0
		60	9,220	+3.0
With	17,850	20	17,540	-1.7
		40	17,020	-4.6
		60	18,820	-0.2

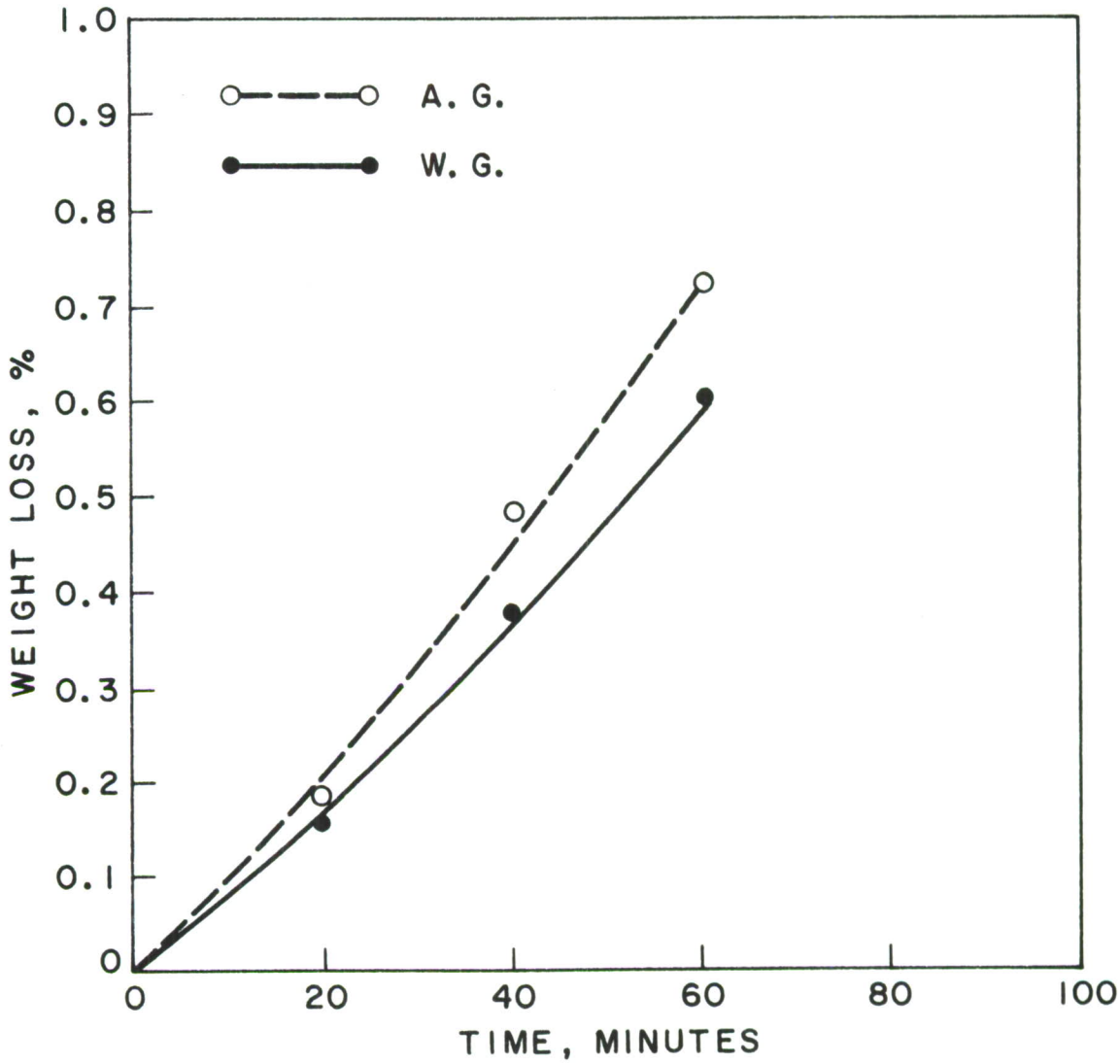


Fig. 24 - WEIGHT CHANGE ON EXPOSURE TO STATIC AIR AT 2000°C AND 10^{-5} TORR PRESSURE, BILLET 7-F-12.

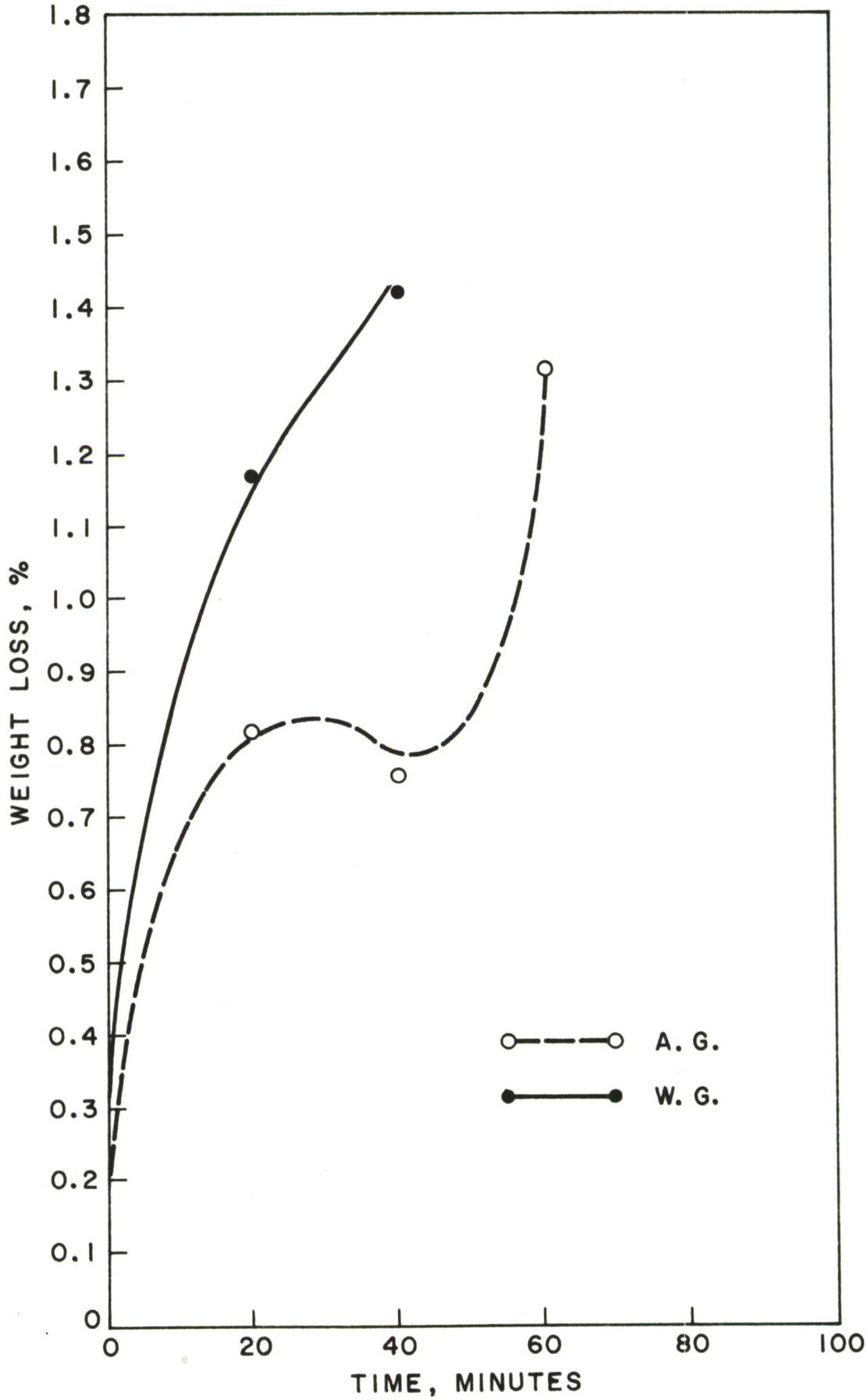


Fig. 25 - WEIGHT CHANGE ON EXPOSURE TO STATIC AIR AT 1000°C AND ATMOSPHERIC PRESSURE, BILLET 7-F-12.

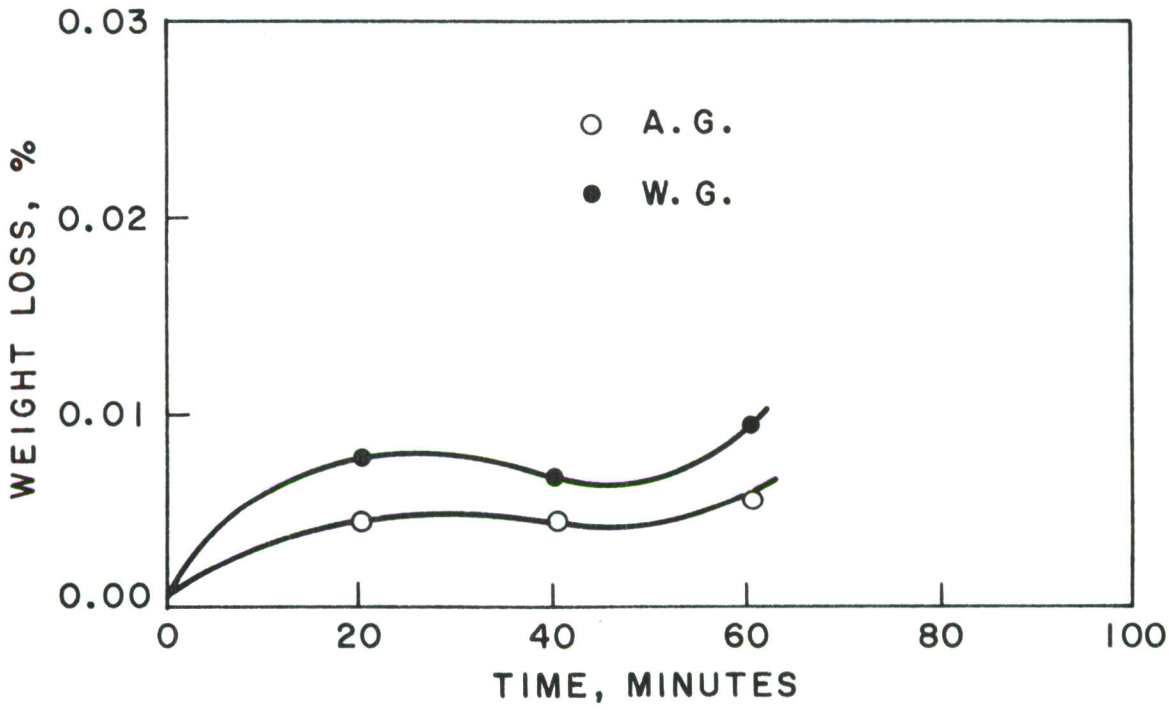


Fig. 26 - WEIGHT CHANGE ON EXPOSURE TO STATIC AIR AT 1000°C AND 10^{-5} TORR PRESSURE, BILLET 7-F-12

weight loss; however, as more and more of the zirconium diboride and silicon carbide are exposed, an oxide coating is formed that would reduce the graphite loss and thus result in a net weight increase. Once the coating has formed, a net weight loss would be observed due to diffusion of oxygen through the protective coating and the loss of graphite as CO and CO_2.

Metallographic analysis of the oxidized surface shows a certain amount of surface attack for the air-exposed specimen and less attack for the material tested in a partial vacuum, Figs. 27 and 28. However, the photomicrographs are inconclusive and a chemical analysis is being made to determine what new products are formed at the surface under oxidizing conditions.

V. SPECIFIC HEAT

Enthalpies of the three JTA graphites were measured from room temperature to 4000°F (2225°C) using the drop-calorimeter technique. A sample of each material was heated to a desired temperature in a furance and then dropped into a modified Parr calorimeter.

Three drops were made at each temperature level for each material in order to assure the accuracy and the reproducibility of the results. The process was repeated at successively higher temperatures at intervals of approximately 200°F (110°C).

The enthalpy of the material was calculated from the experimental measurements using the following equation:

$$\Delta H = \frac{W_e (t_f - t_i)}{M_s}$$

where W_e = water equivalent, receiver, and weight water contained in bucket, in Btu/°F

t_f, t_i = final and initial calorimeter temperatures, respectively, as measured by the calorimeter thermometer, in °F

M_s = mass of sample, in pounds

From the enthalpy values, the enthalpy curve vs. temperature was obtained. An equation was fitted to this enthalpy curve, and specific heat was determined by differentiating it with respect to temperature at constant pressure.

$$C_p = \left(\frac{\partial H}{\partial t}\right)$$

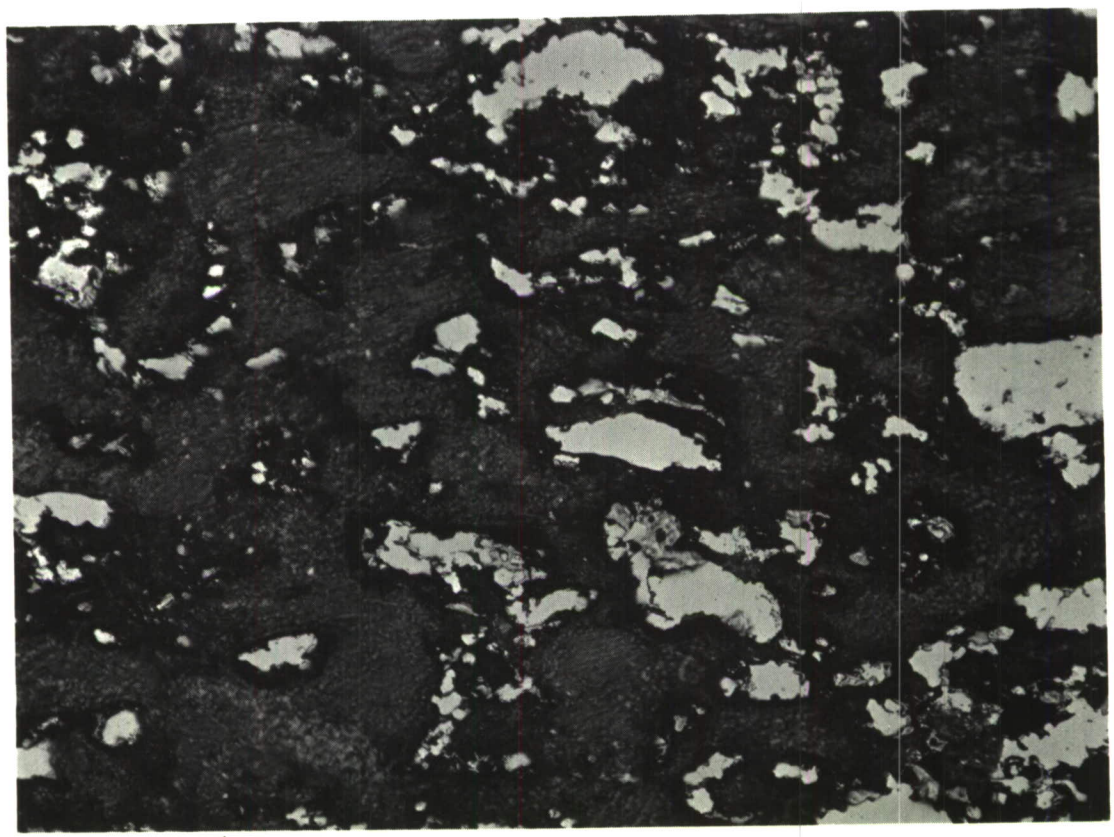
where C_p = specific heat at constant pressure

H = enthalpy

t = temperature

EXTERIOR EDGE

Fig. 27a - PHOTOMICROGRAPHS OF JTA OXIDIZED IN AIR AT 2000°C AND 5 x 10^{-5} TORR PRESSURE FOR 1 HR (320X)

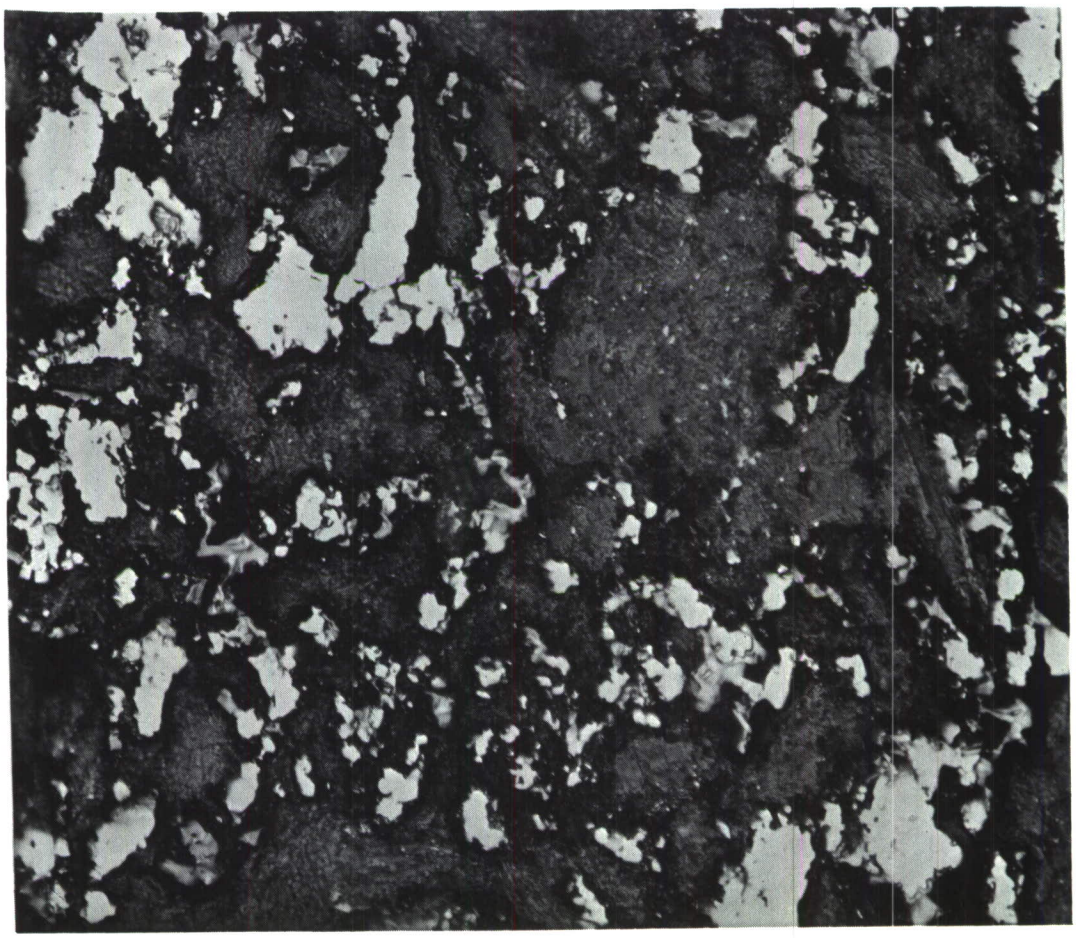


INTERIOR EDGE

Fig. 27b - PHOTOMICROGRAPH OF JTA OXIDIZED IN AIR AT 2000°C AND 5×10^{-5} TORR PRESSURE FOR 1 HR (320 X)

EXTERIOR EDGE

Fig. 28a - PHOTOMICROGRAPH OF JTA OXIDIZED IN AIR AT 1000°C AND 1 ATMOSPHERE FOR 1 HR (320X)



INTERIOR EDGE

Fig. 28b - PHOTOMICROGRAPH OF JTA OXIDIZED IN AIR AT 1000°C AND 1 ATMOSPHERE FOR 1 HR (320X)

The enthalpies of the three materials as functions of temperature are presented in Tables XV through XVII and in Fig. 29.

Since there was no significant difference between enthalpy values of all three JTA graphites, one function for specific heat versus temperature was developed for all three materials. This is shown in Table XVIII and Fig. 30.

VI. REFERENCES

1. S. A. Bortz and R. E. Welch, "Evaluation of Graphite Refractory Composites for High-Temperature Structural Use," IITRI-G6004-1 (Final Report), July 15, 1965.

2. K. V Zeitsch and J. M. Criscione, "Oxidation-Resistant Graphite-Base Composite," WADD-TR-61-72, Vol. XXX, August 1963.

3. P.T.B.Shaffer, High-Temperature Materials, Plenum Press, New York, 1964.

4. "Graphite-Metal Composites," Contract No. NASr-65(09), IITRI Project No. G6003, Quarterly Report No. 3, April 21, 1966.

5. F.M. Anthony, L. Marcus, and A. L Mistretta, "Selection Techniques for Brittle Materials" (Research toward the Evaluation of JTA Graphite Composite as a Structural Refractory Ceramic Body) Contract No. AF 33(615)-2376, Report No. 2178-900601, February 1966.

6. W. A. Wallis and H. V. Roberts, Statistics, A New Approach, Free Press of Glencoe, Inc., Jan. 1962.

Table XV

ENTHALPY VALUES FOR JTA GRAPHITE (7-F-14)

Temperature, °F	ΔH, Btu/lb
80	0
215	21
426	60
630	105
784	145
973	196
1154	245
1342	300
1510	350
1750	425
1912	477
2143	560
2297	605
2510	685
2723	774
2905	845
3120	920
3296	1000
3471	1080
3652	1147
3789	1232
3973	1310

Table XVI

ENTHALPY VALUES FOR JTA GRAPHITE (7-F-12)

Temperature, °F	ΔH, Btu/lb
80	0
178	15
362	48
583	95
730	130
912	180
1098	230
1306	293
1470	338
1620	385
1835	452
2057	528
2210	579
2445	665
2681	750
2901	843
3124	925
3305	1003
3497	1086
3670	1162
3809	1230
3962	1315

Table XVII

ENTHALPY VALUES FOR JTA GRAPHITE (14-G-1)

Temperature, °F	ΔH, Btu/lb
80	0
212	20
415	58
620	103
783	143
963	192
1140	241
1315	290
1508	350
1690	404
1903	475
2160	560
2345	627
2516	690
2749	782
2910	841
3117	922
3315	1005
3491	1080
3663	1154
3796	1221
3971	1312

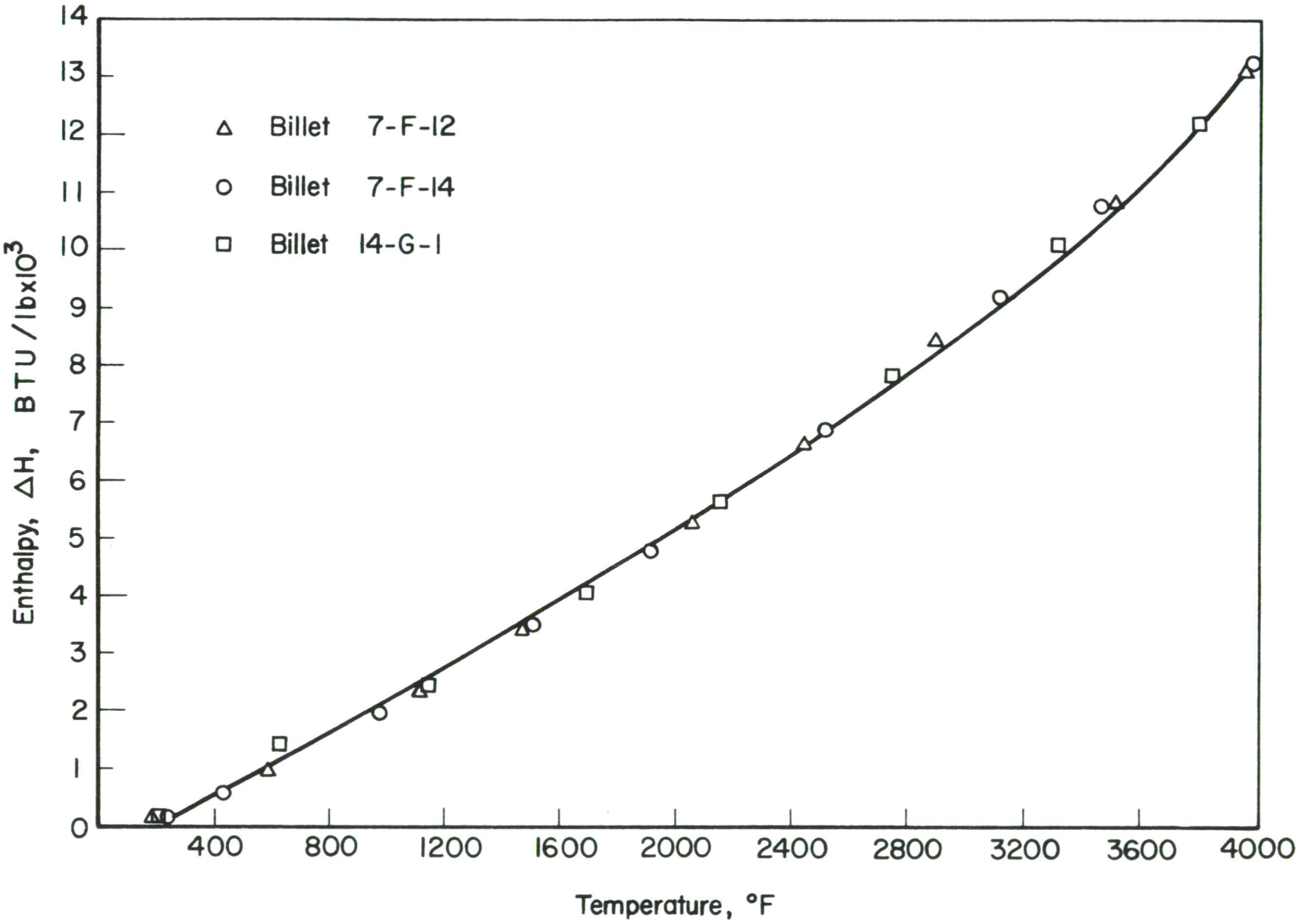


Fig.29 - ENTHALPY OF JTA VS. TEMPERATURE

Table XVIII

SPECIFIC HEAT OF JTA GRAPHITE SPECIMENS

(7-F-14, 7-F-12, 14-G-1)

Temperature, °F	C_p, Btu/lb °F
500	0.220
1000	0.270
1500	0.310
2000	0.325
2500	0.360
3000	0.390
3500	0.430
4000	0.470

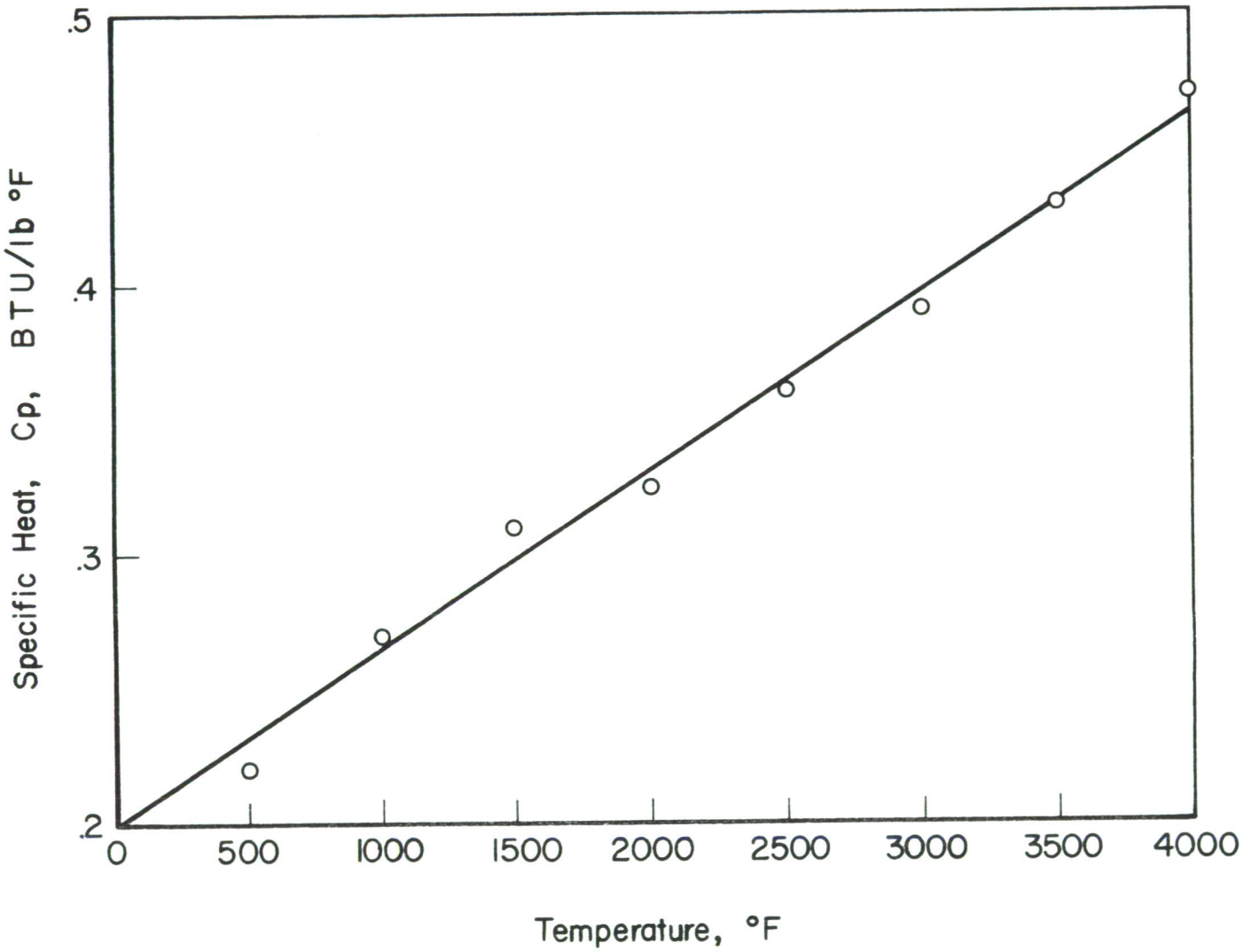


Fig. 30 - SPECIFIC HEAT OF JTA VS. TEMPERATURE

APPENDIX

ANALYSIS OF VARIANCE OF FLEXURAL STRENGTH FOR 7-F-12, 7-F-14, AND 14-G-1

An analysis of variance is a test, whereby one may determine whether or not several samples are members of the same parent population. The test compares the variation of values between members of the samples with the variation between samples. If all of the samples are from the same population, the ratio of these two variations should be small. A large ratio, on the other hand, is an indication that there are differences between samples either in the material or in the test itself.

The following is an outline of the method of calculation used in the analysis:

N = number of items in all samples

T = grand total of all of the values measured

$\bar{X}$ = grand average T/N

n = number of samples

i = number of items in a given sample

$\bar{x}$ = the average of a given sample

To calculate the variance between samples

$$\sum_{n} (\bar{x}_n - \bar{X})^2 i_n = \text{between-samples sum of squares}$$

$n - 1$ = between-samples degrees of freedom, then

$$\frac{\sum_{n} (\bar{x}_n - \bar{X})^2 i_n}{n - 1} = \text{between-samples estimate of variance}$$

To calculate the variance with the samples,

$$\sum_{ni} (x_i - \bar{x}_n)^2 = \text{within-sample sum of squares}$$

$$\sum_{n} (i_n - 1) = \text{within-sample degrees of freedom}$$

then $$\frac{\sum_{ni} (x_i - \bar{x}_n)^2}{\sum_{n} (i_n - 1)} = \text{within-sample estimate of variance.}$$

The largest ratio of these two estimates is then compared with tables which indicate the values of the ratio which will be exceeded with various degrees of probability* (i.e., in the case analyzed, the ratio will exceed a value of 19.5 in 5% of the analyses and will exceed 99 in only 1% of them). Normally, a calculated value which exceeds the 1% value from statistical tables is taken as a very strong indication of real differences between samples and a value exceeding the 5% value is taken as a moderate indication of real differences between samples.

As may be seen from Tables XIX to XXI, in the analysis carried out on JTA specimens, all tests except the sonic modulus test give variance ratios well below the 5% level. The high values of the ratio obtained in the case of sonic modulus, when considered in the light of the low values obtained in the flexure and tangent modulus analyses, seem to indicate a variability in the test rather than in the material.

---

* Moroney, M. J., Facts From Figures, Penguin Books, Baltimore (1954).

Table XIX

ANALYSIS OF VARIANCE FOR FLEXURE STRENGTHS
OF 3 x 1/4 x 1/4 IN. SPECIMENS
FROM BILLETS 7-F-12, 7-F-14, AND 14-G-1

Source of Variation	Sums of Squares	Degrees of Freedom	Variance Estimate	F*
	With Grain			
Between samples	5,338,040	2	2,669,020	2.86
Within samples	206,067,650	27	7,632,135	
Total	211,405,690	29		
	Across Grain			
Between samples	9,271,710	2	4,635,855	9.3
Within samples	13,479,100	27	498,070	
Total	22,750,810	29		

* 1% level of F = 99; 5% level of F = 19.5.

Table XX

ANALYSIS OF VARIANCE FOR TANGENT MODULUS
OF 3 x 1/4 x 1/4 IN. SPECIMENS
FROM BILLETS 7-F-12, 7-F-14, AND 14-G-1

Source of Variation	Sums of Squares, $x10^{12}$	Degrees of Freedom	Variance Estimate $x10^{12}$	F*
		With Grain		
Between samples	53.789	2	26.8945	6.78
Within samples	107.085	27	3.966	
Total	160.874	29		
		Across Grain		
Between samples	4.561	2	2.2805	1.67
Within samples	36.752	27	1.361	
Total	41.313	29		

* 1% level of F = 99; 5% level of F = 19.5.

Table XXI

ANALYSIS OF VARIANCE FOR SONIC MODULUS
OF 3 x 1/4 x 1/4 IN. SPECIMENS
FROM BILLETS 7-F-12, 7-F-14, AND 14-G-1

Source of Variation	Sums of Squares $\times 10^{12}$	Degrees of Freedom	Variance Estimate $\times 10^{12}$	F*
		With Grain		
Between samples	25.585	2	12.7925	38.9
Within samples	8.904	27	0.329	
Total	34.489	29		
		Across Grain		
Between samples	415.373	2	207.6865	1070
Within samples	5.215	27	0.193	
Total	420.588	29		

Unclassified
Security Classification

DOCUMENT CONTROL DATA - R&D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1. ORIGINATING ACTIVITY *(Corporate author)*
IIT Research Institute
Chicago, Illinois 60616

2a. REPORT SECURITY CLASSIFICATION
Unclassified

2b. GROUP
None

3. REPORT TITLE
PHYSICAL PROPERTIES OF REFRACTORY MATERIALS

4. DESCRIPTIVE NOTES *(Type of report and inclusive dates)*
Annual Summary, May 1965 to May 1966

5. AUTHOR(S) *(Last name, first name, initial)*
Bortz, Seymour A.

6. REPORT DATE	7a. TOTAL NO. OF PAGES	7b. NO. OF REFS
August 1966	65	6

8a. CONTRACT OR GRANT NO.	9a. ORIGINATOR'S REPORT NUMBER(S)
AF 33(615)-3028	AFML-TR-66-223
b. PROJECT NO. 7381	
c. Task No. 738106	9b. OTHER REPORT NO(S) *(Any other numbers that may be assigned this report)* IITRI-G6001-12
d.	

10. AVAILABILITY/LIMITATION NOTICES

11. SUPPLEMENTARY NOTES	12. SPONSORING MILITARY ACTIVITY
	AFML (MAAM) Wright-Patterson AFB, Ohio

13. ABSTRACT The purpose of this program is to characterize and correlate the physical and mechanical properties of JTA graphite and from these data, determine its critical engineering properties.

Three JTA billets were studied, two of 7 in. diameter by 6 1/2 in. long and one 14 in. diameter by 7 in. long. Examination of gross flaws and differences in density was done using radiographic methods. Densities of the three billets are above 3.00 g/cc. Metallographic, x-ray, and microprobe analyses confirm the presence of graphite, ZrB_2, and βSiC.

Mechanical measurements indicate that JTA graphite is anisotropic; the "with" grain orientation is about twice as strong as the "across" grain orientation. Size effect studies indicate that a minimum 1/4 in. cross-section dimension is adequate for determining average mechanical properties. Studies were made using both flexure and tension measurements. The mechanical strength of the three billets appears to be the same.

Temperature and oxygen pressure have a great effect on the oxidation resistance of JTA graphite. The material appears to be more resistant at the higher temperatures.

DD FORM 1 JAN 64 1473

Unclassified
Security Classification

14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Characterize and correlate physical and mechanical properties. Two 7" billet; one 14" billet. High temperature testing in inert and oxidizing atmospheres.						

INSTRUCTIONS

1. ORIGINATING ACTIVITY: Enter the name and address of the contractor, subcontractor, grantee, Department of Defense activity or other organization *(corporate author)* issuing the report.

2*a*. REPORT SECURITY CLASSIFICATION: Enter the overall security classification of the report. Indicate whether "Restricted Data" is included. Marking is to be in accordance with appropriate security regulations.

2*b*. GROUP: Automatic downgrading is specified in DoD Directive 5200.10 and Armed Forces Industrial Manual. Enter the group number. Also, when applicable, show that optional markings have been used for Group 3 and Group 4 as authorized.

3. REPORT TITLE: Enter the complete report title in all capital letters. Titles in all cases should be unclassified. If a meaningful title cannot be selected without classification, show title classification in all capitals in parenthesis immediately following the title.

4. DESCRIPTIVE NOTES: If appropriate, enter the type of report, e.g., interim, progress, summary, annual, or final. Give the inclusive dates when a specific reporting period is covered.

5. AUTHOR(S): Enter the name(s) of author(s) as shown on or in the report. Enter last name, first name, middle initial. If military, show rank and branch of service. The name of the principal author is an absolute minimum requirement.

6. REPORT DATE: Enter the date of the report as day, month, year; or month, year. If more than one date appears on the report, use date of publication.

7*a*. TOTAL NUMBER OF PAGES: The total page count should follow normal pagination procedures, i.e., enter the number of pages containing information.

7*b*. NUMBER OF REFERENCES: Enter the total number of references cited in the report.

8*a*. CONTRACT OR GRANT NUMBER: If appropriate, enter the applicable number of the contract or grant under which the report was written.

8*b*, 8*c*, & 8*d*. PROJECT NUMBER: Enter the appropriate military department identification, such as project number, subproject number, system numbers, task number, etc.

9*a*. ORIGINATOR'S REPORT NUMBER(S): Enter the official report number by which the document will be identified and controlled by the originating activity. This number must be unique to this report.

9*b*. OTHER REPORT NUMBER(S): If the report has been assigned any other report numbers *(either by the originator or by the sponsor)*, also enter this number(s).

10. AVAILABILITY/LIMITATION NOTICES: Enter any limitations on further dissemination of the report, other than those imposed by security classification, using standard statements such as:

(1) "Qualified requesters may obtain copies of this report from DDC."

(2) "Foreign announcement and dissemination of this report by DDC is not authorized."

(3) "U. S. Government agencies may obtain copies of this report directly from DDC. Other qualified DDC users shall request through _____."

(4) "U. S. military agencies may obtain copies of this report directly from DDC. Other qualified users shall request through _____."

(5) "All distribution of this report is controlled. Qualified DDC users shall request through _____."

If the report has been furnished to the Office of Technical Services, Department of Commerce, for sale to the public, indicate this fact and enter the price, if known.

11. SUPPLEMENTARY NOTES: Use for additional explanatory notes.

12. SPONSORING MILITARY ACTIVITY: Enter the name of the departmental project office or laboratory sponsoring *(paying for)* the research and development. Include address.

13. ABSTRACT: Enter an abstract giving a brief and factual summary of the document indicative of the report, even though it may also appear elsewhere in the body of the technical report. If additional space is required, a continuation sheet shall be attached.

It is highly desirable that the abstract of classified reports be unclassified. Each paragraph of the abstract shall end with an indication of the military security classification of the information in the paragraph, represented as *(TS)*, *(S)*, *(C)*, or *(U)*.

There is no limitation on the length of the abstract. However, the suggested length is from 150 to 225 words.

14. KEY WORDS: Key words are technically meaningful terms or short phrases that characterize a report and may be used as index entries for cataloging the report. Key words must be selected so that no security classification is required. Identifiers, such as equipment model designation, trade name, military project code name, geographic location, may be used as key words but will be followed by an indication of technical context. The assignment of links, rules, and weights is optional.